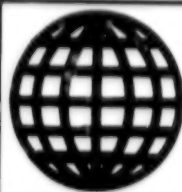


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# ***JPRS Report***

# **Science & Technology**

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***USSR: Chemistry***

# Science & Technology

## USSR: Chemistry

JPRS-UCH-90-008

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20 September 1990

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UDC 541.128+546.943.7

**Influence of Method of Producing Pd/MgO Catalysts on Their Activity in Oxidation of Carbon Monoxide**

907M0113A Moscow ZHURNAL FIZICHESKOY KHMII in Russian Vol 63 No 12, Dec 89 (Manuscript received 22 Jun 88) pp 3208-3213

[Article by N. I. Ivashchenko, L. S. Kravchuk, P. V. Kurman, I. P. Mardilovich, and V. L. Shirokiy, Institute of Physical-Organic Chemistry, Belorussian Academy of Sciences, Minsk]

[Abstract] The oxidation of CO in air containing 1 percent CO at atmospheric pressure on Pd/MgO specimens is studied. The change in phase composition, concentration, paramagnetic properties of Pd<sup>+</sup>, and redox properties is studied as a function of the method of production and palladium content. Under the experimental conditions, which are rather close to real catalysis, the temperature of full consumption of carbon monoxide is found to be independent of the Pd<sup>+</sup> ion concentration, which is stabilized by the magnesium oxide lattice. Therefore, specimens of hydroxides with Pd<sup>+</sup> ions contained in the MgO lattice at low metal concentrations are significantly less active in the oxidation of CO than are catalysts in which the metal is more independent of the carrier, such as specimens obtained by saturation of crumbled magnesium oxide preliminarily heated at 1,070 K in air with a solution of bis- $\pi$ -allyl palladium chloride in dry benzene, stored over NaOH, and heated to 770 K immediately before the experiments. Figures 2; References 15: 10 Russian, 5 Western.

UDC 541.128

**Catalytic Condensation of (-) Epicatechin With Iron Ions Present**

907M0113B Moscow ZHURNAL FIZICHESKOY KHMII in Russian Vol 63 No 12, Dec 89 (Manuscript received 22 Aug 88) pp 3214-3220

[Article by S. O. Travin, G. G. Duka, and A. P. Purmal, Institute of Chemical Physics, USSR Academy of Sciences, Moscow]

[Abstract] The detailed mechanism of the interaction of epicatechin and other polyphenols with metal complexes is studied by the interrupted-jet method in the presence of iron ions. The kinetic curve indicates that it is a first-order reaction, while the concentration variation indicates an order higher than first. The contradiction is resolved by assuming that the decrease in concentration of the intermediate complex formed is not accompanied by a change in total concentration of trivalent iron, but rather is caused by consumption of epicatechin, so that the substrate is expended nonstoichiometrically in the presence of a smaller concentration of Fe(III). The rate constants of the formation and decomposition of the

intermediate charge-transfer complex are computed. Although unambiguous identification of the intermediate radical products is impossible, the system suggested here describes all of the observed kinetic regularities of the consumption of epicatechin in the presence of iron ions. Figures 5; References 6: Russian.

UDC 541.128

**Interstitial Graphite Compounds and Their Catalytic Activity in Decomposition of Ozone**

907M0113C Moscow ZHURNAL FIZICHESKOY KHMII in Russian Vol 63 No 12, Dec 89 (Manuscript received 3 Oct 88) pp 3228-3232

[Article by S. V. Osintseva, L. Ye. Gorlenko, G. I. Yemelyanova, L. D. Kvacheva, Yu. N. Novikov, and M. Ye. Volpin, Moscow State University imeni M. V. Lomonosov; Institute of Heteroorganic Compounds, Moscow]

[Abstract] A comparative study is presented of the catalytic properties of layered compounds of graphite with the chlorides of Pd(II), Co(II), Cu(II), Fe(II), Fe(III), Ni(II), Zn(II), and Mo(V). All of the catalysts are found to operate stably for long periods of time. The time required to reach the steady state depends on the nature of the interstitial substance and the initial temperature of the experiment. The greatest activity is that of catalysts with PdCl<sub>2</sub> and CoCl<sub>2</sub>. The kinetic parameters of the process are determined, and interstitial compounds are found to have an advantage over surface-applied catalysts. The activity of the compounds is generally higher than that of the salts alone or of the salts applied to graphite. Figures 3; References 13: 12 Russian, 1 Western.

UDC 537.525.539.23

**Change in Surface of Silicon in Activated Nitrogen Pulse Discharge**

907M0113D Moscow ZHURNAL FIZICHESKOY KHMII in Russian Vol 63 No 12, Dec 89 (Manuscript received 27 Apr 89) pp 3362-3367

[Article by O. M. Knipovich, N. I. Konyushkina, V. I. Voronina, and L. I. Nekrasov, Moscow State University imeni M. V. Lomonosov]

[Abstract] Measurements of the polarization angles  $\psi$  and  $\Delta$  were performed on a type LEF-ZM ellipsometer at 6328 Å with incident angles of 70, 72, and 74°. The index of refraction and surface film thickness were determined by numerical solution of an equation system representing a model of a transparent film on a silicon substrate with known optical constants. It is found that at 600-700°C the surface of the silicon is oxidized due to activation of oxidizer impurities in the nitrogen in the discharge. It is found that at 900°C thin layers containing silicon nitride are formed on the surface of the silicon. References 9: 4 Russian, 5 Western.

UDC 541.128.5

**Determination of Order of Catalytic Reactions from Concentration of Active Catalyst Centers**

907M0114A Moscow VESTNIK MOSKOVSKOGO UNIVERSITETA: KHIMIYA in Russian Vol 30 No 6, Nov-Dec 89 Seriya 2 (Manuscript received 29 Apr 88) pp 557-561

[Article by Yu. I. Azimova and R. Ye. Mardaleysvili]

[Abstract] The authors have developed a new version of a method for determining the sequence of catalytic reactions based on the concentration of active centers by processing experimental data obtained with a single specimen. The processing of experimental data is based on direct observation of the process leading to poisoning of the catalyst and determination of the rate of that process. Examples are presented that demonstrate the capability of the method to develop formal kinetic information on the order of the main reaction and the process of catalyst poisoning based on the concentration of active centers for various processes of conversion of hydrocarbons on both oxide and metallic catalysts. Figures 4; References 15: 4 Russian, 11 Western.

UDC 541.138

**Preparation and Electrocatalytic Properties of Palladium-Phosphorus Alloy**

907M0114B Moscow VESTNIK MOSKOVSKOGO UNIVERSITETA: KHIMIYA in Russian Vol 30 No 6, Nov-Dec 89 Seriya 2 (Manuscript received 29 Jun 88) pp 562-566

[Article by N. S. Kopylova and O. A. Petriy]

[Abstract] The authors have produced the alloy PdP by chemical and electrochemical precipitation. The volumetric composition of the microcrystalline alloy was determined by local x-ray spectral analysis to be 87.0 at. percent Pd, 13.0 percent P. The studies indicated that the mechanism of oxidation of methanol, formic acid, and formaldehyde on the PdP alloy was the same as on disperse Pd. Both microcrystalline and crystalline specimens of the alloy have identical activity in all the reactions studied. The addition of phosphorus thus does not improve the electrocatalytic properties of the palladium. Figures 2; References 16: 7 Russian, 9 Western.

UDC 541.12.036:546.97

**Influence of Heat Treatment Conditions on Properties of Electroprecipitated Rhodium**

907M0114C Moscow VESTNIK MOSKOVSKOGO UNIVERSITETA: KHIMIYA in Russian Vol 30 No 6, Nov-Dec 89 Seriya 2 (Manuscript received 1 Jul 88) pp 584-588

[Article by L. P. Mashkova and A. I. Pletyushkina]

[Abstract] Experimental data are presented on the influence of heat treatment at 100-600°C in various gas atmospheres and in a vacuum on the adsorption and catalytic properties of electroprecipitated rhodium in order to differentiate the influence of temperature and the medium in which the heat treatment occurs. It is found that inert atmospheres act to soften the effect of the heat treatment in comparison to a vacuum. The vacuum causes the electroprecipitated substance to be more compact. Aggregates of particles become larger in an argon atmosphere. In hydrogen, needle formations appear, and the electroprecipitate is dispersed. Figures 4; References 12: Russian.

UDC 547.535.1:66.094.187.3

**Efficiency of Fe/Cr/K Catalysts in Propylbenzene Dehydrogenation**

907M0192A Moscow NEFTEKHIMIYA in Russian Vol 30 No 1, Jan-Feb 90 (manuscript received 5 Jul 89) pp 22-25

[Article by B. Yu. Smirnov, O. I. Kondratyev, V. S. Safronov, and G. Ya. Bogomolova, Kuybyshev Polytechnical Institute imeni V. V. Kuybyshev]

[Abstract] Trials were conducted with a series of Fe/Cr/K catalysts to assess their suitability for dehydrogenation of propylbenzenes, as well as to determine the effects of hydrogen and carbon dioxide on the system. In the case of isopropylbenzene dehydrogenated at 773-823 K, the commercial catalysts ranked as follows: D-71 > K-22 > KMS. Studies on the dehydrogenation of p-propylbenzene yielded the following ranking of the tested catalysts: D-71 > K-22 > K-24 > KMS > KS-4. Introduction of hydrogen or carbon dioxide into the system depressed the rate of dehydrogenation; in the case of hydrogen, however, selectivity was improved. Tables 4; references 12: 11 Russian, 1 Western.

UDC 547.52:542.941.7:546.74

**Hydrogenation of Benzene and Toluene on Ni Catalysts Modified With Heteropolytungstates**

907M0192B Moscow NEFTEKHIMIYA in Russian Vol 30 No 1, Jan-Feb 90 (manuscript received 9 Jun 89) pp 26-29

[Article by M. D. Navalikhina, I. L. Malkina, and V. I. Garanin, Institute of High Temperatures, USSR Academy of Sciences, Moscow]

[Abstract] An analysis was conducted of the efficiency of Ni catalysts in the hydrogenation of benzene and toluene in relation to Ni concentration and doping of the Al<sub>2</sub>O<sub>3</sub> support with heteropolytungstates (HPTs). The resultant kinetic data demonstrated that catalysts of this type, containing 1-4 percent Al by weight and HPT (including some incorporating Co and Cr), exceeded the activities of more conventional catalysts containing 8-50 percent Al by weight. Catalytic activity was shown to increase



linearly with an increase in the Ni content from 1-4 percent with the use of simple HPTs. In the case of the various HPTs the hydrogenation curves went through a maximum. Accordingly, the results demonstrated that targeted catalysts may be designed by varying the Ni/HPT ratio. Figures 2; tables 2; references 7 (Russian).

UDC 547.211:662.763:542.962

#### **Coupled Conversion of Methane and Carbon Dioxide on Mn Catalysts**

907M0192C Moscow NEFTEKHIMIYA in Russian  
Vol 30 No 1, Jan-Feb 90 (manuscript received  
30 May 89) pp 30-34

[Article by A. Kh. Mamedov, S. R. Mirzabekova, Sh. A. Nuriyev, P. A. Shiryayev and D. P. Shashkin, INKhP [expansion unknown], Baku; Chemical Physics Institute, USSR Academy of Sciences, Moscow]

[Abstract] A comparative assessment was conducted of the coupled conversion of methane and carbon dioxide on Mn-Ca/Al<sub>2</sub>O<sub>3</sub>, Mn-Ba/Al<sub>2</sub>O<sub>3</sub>, and Ni-Ca/Al<sub>2</sub>O<sub>3</sub> catalysts at 840-980°. The resultant data showed that on the Mn catalysts both the forward and reverse reactions proceeded without the formation of carbide deposits on the catalysts, a problem encountered with the Ni catalyst. As a result, with equimolar concentrations of methane and carbon dioxide, a stable conversion process prevails with Mn catalysts. In the case of the Ni catalyst carbon products are deposited on the catalyst, entailing its disruption and inactivation. Figures 2; tables 1; references 8: 6 Russian, 2 Western.

UDC 547.214:665.652.4:661.183.6

#### **Alkylation of Isobutane With Ethene on Zeolite Catalyst PdCaRZENU**

907M0192D Moscow NEFTEKHIMIYA in Russian  
Vol 30 No 1, Jan-Feb 90 (manuscript received 2 Jan 89)  
pp 35-39

[Article by M. A. Timurziyeva, S. N. Khadzhiev, Mariyam A. Timurziyeva, V. L. Bayburskiy, and I. L. Aleksandrova, Grodno Petroleum Scientific Research Institute]

[Abstract] Zeolite PdCaRZNU-catalyzed alkylation of isobutane by ethene was monitored over a wide range of experimental conditions to determine the optimum conditions for the ethene conversion process. The resultant findings demonstrated that the following represented the best reaction parameters: 323-363 K, 0.7-0.8 hours' contact time, and 17:1 isobutane:ethene ratio. In the case of complete ethene conversion the yield of alkyl benzenes was 203.7-217.6 percent by weight, with C-8 isoparaffins representing 60.0 to 68.7 percent of the total. Polymerization, with a concomitant reduction in the alkyl benzene yield, was promoted by elevation of the temperature, a decrease in the isobutane:ethene ratio, and reduction of the contact time. The reduction in yield

was attributed to the deposition of condensation products on the catalyst, thereby reducing its efficiency. Finally, reduction in the isobutane:ethene ratio was shown to favor an increase of the C-9+ fraction and oligomeric C-6 and C-8 components in the product. Figures 1; tables 2; references 6 (Russian).

UDC 542.973:66.092

#### **Platinum Promoters and Regeneration of Pellet-Type Zeolite Cracking Catalyst**

907M0192E Moscow NEFTEKHIMIYA in Russian  
Vol 30 No 1, Jan-Feb 90 (manuscript received 3 Oct 89)  
pp 40-45

[Article by N. A. Zakarina, G. D. Zakumbayeva, E. N. Litvyakova, M. I. Levinbuk, and Kh. K. Magomadova, Institute of Organic Synthesis and Electrochemistry imeni D. V. Sokolskiy, Kazakh SSR Academy of Sciences; Grodno Petroleum Scientific Research Institute]

[Abstract] Studies were conducted on the efficiency of trace quantities of Pt promoters in zeolite-based pellet-type catalysts used for cracking kerosene-gas oil fractions. Studies with Zeokar-2 catalyst demonstrated that promotion with 1 to 100 ml<sup>-1</sup> Pt increased the CO<sub>2</sub>/CO ratio from a control value of 0.9 to 6.5-12.1 at 733 K. The highest rate of CO oxidation was obtained at 10 ml<sup>-1</sup> Pt. In addition, catalysts promoted with Pt<sup>0</sup> exhibited much greater stability than did those promoted with Pt<sup>4+</sup>. This difference was attributed to the fact that in the case of the Pt<sup>0</sup> catalysts significantly less of the Al<sub>2</sub>Pt phase was formed and, accordingly, dispersity of Pt was not as adversely affected as in the Pt<sup>4+</sup>-promoted catalysts. Figures 3; tables 2; references 6: 4 Russian, 2 Western.

UDC 547.593.057:542.938

#### **Hydrolytic Transformation of Cyclohexylamine Into Cyclohexanone and Cyclohexanol on Ni/Cu/Cr Catalyst**

907M0192F Moscow NEFTEKHIMIYA in Russian  
Vol 30 No 1, Jan-Feb 90 (manuscript received  
24 Feb 89) pp 73-77

[Article by Ye. D. Mikhilchenko, B. Z. Fridman, and V. V. Nikitenko, Kuzbass Polytechnical Institute, Kemerovo]

[Abstract] An analysis was conducted on the hydrolysis of cyclohexylamine on Ni/Cu/Cr catalysts to ascertain the sequence leading to the formation of cyclohexanone and cyclohexanol. The combination of kinetic and analytical techniques employed demonstrated that at 175-225°C cyclohexanone is formed directly from cyclohexylamine, along with NH<sub>3</sub> and H<sub>2</sub>. Subsequently, cyclohexanone is hydrogenated by the H<sub>2</sub> to cyclohexanol. Accordingly, an intermediate cyclohexylimine form appears not to be involved. Figures 3; tables 1; references 4 (Russian).



UDC 543.257.2:547.241

**Calcium-Selective Electrodes From Didentate Organophosphorus Compounds**

907M0120A Ivanovo IZVESTIYA VYSSHIKH  
UCHEBNYKH ZAVEDENIY: KHIMIYA I  
KHIMICHESKAYA TEKHOLOGIYA in Russian  
Vol 32 No 2, Sep 89 (manuscript received  
18 Jul 88) pp 46-49

[Article by O. M. Petrukhin, G. V. Bodrin, Yu. M. Polikarpov, and M. I. Kabachnik, Chemical Technology Institute imeni D. I. Mendeleyev, Moscow; Elementorganic Compounds Institute, Moscow]

[Abstract] Previous study of the electrochemical of various diphosphine dioxide electrodes selective to calcium ions shows that using the o-xylylene group as a bridge in diphosphinedioxides makes it possible to increase its selectivity to alkaline and alkaline-earth metal ions. Introduction of electron-donor substituents to the phosphorus-tolyl groups also raises the selectivity of the ligand to calcium in comparison to alkaline and alkaline-earth elements. The complex-forming properties of polydentate phosphorus-containing compounds depends greatly on the mutual distribution of the phosphoryl groups. In the present work a study was made of a number of bidentate organophosphorus compounds as electrode-active substances in calcium-selective electrodes. It was demonstrated that an electrode based on tetratolyl-m-xylylenediphosphine dioxide manifests high selectivity to magnesium ions as compared to all other previously studied electrodes and that this electrode might have certain practical applications. References 6: 5 Russian, 1 Western.

UDC 661.634.2:631.851

**Optimizing the Derivation Process of Extraction Grade Phosphoric Acid From Karatau Phosphorites Using the Dihydrate-Semihydrate Method**

907M0120B Ivanovo IZVESTIYA VYSSHIKH  
UCHEBNYKH ZAVEDENIY: KHIMIYA I  
KHIMICHESKAYA TEKHOLOGIYA in Russian  
Vol 32 No 2, Sep 89 (manuscript received  
12 Aug 88) pp 60-63

[Article by I. A. Petropavlovskiy, S. L. Akhnazarova, O. V. Kuznetsova, and O. A. Strashnenko, Chemical Technology Institute imeni D. I. Mendeleyev, Moscow]

[Abstract] Ore from the Karatau deposit used to make extraction grade phosphoric acid has a low phosphorus pentoxide content (24-25 percent) and a high magnesium content (up to 5 percent MgO). The dihydrate method employed in refining the Karatau phosphorite ore gives a product having a phosphorus pentoxide content of 20-22 percent. Using this acid to produce fertilizer entails high energy requirements for evaporation. A previous study demonstrated that the dihydrate-semihydrate method

gives a product having a higher concentration of phosphorus pentoxide. In the first stage only part of the stoichiometric portion of sulfuric acid is used to provide primary crystallization of calcium sulfate dihydrate. Recrystallization of the latter into calcium sulfate semihydrate in the second stage takes place at a higher  $\text{SO}_3$  and  $\text{P}_2\text{O}_5$  concentration in the slurry. This frees some of the phosphorus pentoxide entrapped in the residue. In the present work optimum conditions for running this process were determined using mathematical methods for planning and generalization of indicators (the Harrington function). The results demonstrate that the dihydrate-semihydrate method could be effectively used to give a product with phosphorus pentoxide-to-acid yield of 96.5-97 percent. The relatively low phosphorus pentoxide content in the residue (to 1 percent) makes it possible to use this product as a building material. Figures 2; references 5: 4 Russian, 1 Western.

UDC 66.063.8:661.632.11:66.023

**Mixing Phosphoric Acid Slurry in Various Size Reactors with Agitators**

907M0120C Ivanovo IZVESTIYA VYSSHIKH  
UCHEBNYKH ZAVEDENIY: KHIMIYA I  
KHIMICHESKAYA TEKHOLOGIYA in Russian  
Vol 32 No 2, Sep 89 (manuscript received  
4 Jul 88) pp 94-97

[Article by A. V. Bepalov, V. A. Posokhov, and V. S. Beskov, Chemical Technology Institute, imeni D. I. Mendeleyev, Moscow]

[Abstract] During sulfuric acid refining of natural phosphates into extraction grade phosphoric acid, the phosphoric acid slurry becomes homogenized through mixing in the working zone of the reactor, i.e. the temperature and concentration gradients are lowered. Although much has been published on the problems of mixing suspensions with agitators, the problem of scaler transition from a large mixing volume (as in industrial units) to small (laboratory models) and vice versa, has not yet been finalized. This is primarily due to the fact that for most liquid-solid mixing systems, it is not possible to reach full homogeneity throughout the entire working volume. Analysis of published data shows the lack of an invariant criterion for scaler transition for mixing suspensions in various size reactors, and it becomes necessary to observe the conditions of geometric and hydrodynamic behavior, also noting the linear nature of the relationship between the energy expended on mixing the slurry and the scaler value. Comparison of mixing conditions between a laboratory scale (8 and 4 liter) continuous type phosphoric acid reactor with an industrial scale ( $740 \text{ m}^3$ ) unit demonstrates that both the reproducibility of qualitative data on the laboratory model, and parity of specific energy consumption are required for scaler transition. Figures 2; references 10: 3 Russian, 7 Western.

UDC 548.55

### Technology for Growing Potassium Dihydrophosphate Single Crystals

907M0120D Ivanovo IZVESTIYA VYSSHIKH  
UCHEBNYKH ZAVEDENIY: KHIMIYA I  
KHIMICHESKAYA TEKHOLOGIYA in Russian  
Vol 32 No 2, Sep 89 (manuscript received  
12 Aug 88) pp 103-104

[Article by V. G. Vaydanich, Yu. Yu. Firtsak, and I. I. Zimomrya, Uzhgorod State University]

[Abstract] According to published data potassium dihydrophosphate single crystals are grown from aqueous solutions at constant temperature under helium. Under these conditions it is not possible to regulate the growth rate of a single crystal by changing its displacement rate. At constant temperature crystals may also be prepared by absorption of water vapors with deliquescent substances such as calcium chloride or sulfuric acid. In the present work potassium dihydrate single crystals were grown from a molecular solution at constant temperature void of the above difficulties. A crystal seed placed on a crystal carrier was rotated coaxially with a container of deliquescent in a hermetically sealed potassium dihydrophosphate solution. Since thermal contact is maintained between the deliquescent and the solution, temperature stabilization and therefore, saturation, occurs. As the temperature of the solution rises, its saturation value decreases. Figure 1; references 2: 1 Russian, 1 Western.

### Instead of a Pill

907M0148A Moscow RABOCHAYA TRIBUNA  
in Russian No 36, 13 Feb 1990 p 4

[Article by T. Krivtsova: "Instead of a Pill"]

[Text] Nitroglycerine, a drug that cuts off angina pectoris attacks, has a serious competitor. This drug does not simply cut off pain; it prevents it. It is not necessary to swallow this pill. A thin wafer attached to the gum prevents pain for 2-4 hours while protection provided by a nitroglycerine tablet stops after 30 minutes.

The new long-lasting pain killer, developed in laboratories of the Department of Medicinal Polymers, All-Union Scientific Research Institute of Medical Technology, is called trinitrolong.

The prolonged effect of the new drug is not its only advantage over nitroglycerine, says one of the creators of trinitrolong, head of laboratories, candidate of chemical sciences G. Khromov. The attending physician can select the dosage with consideration of the individual characteristics of the patient. The uniform entry of nitroglycerine into the organism and the use of an optimum dose reduce the side effects experienced during use of nitroglycerine. The wafers attach readily to the mucosal surface and, dissolving gradually, distribute the medicine uniformly.

How did the idea of packing medicine on a polymer base arise?

Cosmonauts came to us for assistance. Their work, understandably, is dusty and they develop eye infections frequently. When such infections occur on Earth, they usually do not cause any special problems. Eye drops, prescribed by a physician, can be used several times a day. However, under conditions of weightlessness, it is no simple matter to drop medicine into the eyes. At this point, the first drug on a polymer base appeared. Today, such eye medicine is found in the medicine chest of every cosmonaut. It is also needed on Earth.

The small, thin wafer is placed cautiously under the eyelid. It dissolves in an hour but the effect of the medicine lasts for several more hours. When using eyedrops, only 30-40 percent of the medicine enters the eye; use of the wafer ensures entry of 100 percent and this greatly accelerates the recovery process.

It seems astonishing, but veterinarians are interested in eye medicine prepared on a polymer base. We wonder why a cow would be interested in good vision; she does not read books. However, when a cow suffers any pain or is simply distressed or restless, her milk yield and weight gain drop. Can we prescribe eyedrops for her? What cow would permit this procedure readily? With use of the wafer there are no special problems.

I do not want to delude the reader by speaking about the invention as if it were a medical sensation. These drugs were developed over a period of time and work is now underway in G. Khromov's laboratory on similar medicines for use in other regions of medicine. They are not yet available in drugstores.

The main obstacle to production of the copolymer is the shortage of raw material. Production itself is low-tonnage production but the product must be of high-purity. This is unsuitable for larger enterprises and small enterprises cannot satisfy consumer demand.

Will we see trinitrolong or eye medicine on wafers generally in drugstores? Possibly, you will see them but they will be in packages of an American-Belgium firm. This firm recently bought a license from the Soviet Union and this gives them the right to produce these most marvelous drugs.

It has now become a sad tradition to reap the fruits of our own discoveries through a foreign country.

### Chemical Plant Changes Its Profile

907M0148B Moscow RABOCHAYA TRIBUNA  
in Russian No 40, 13 Feb 1990 p 1

[Article by TASS: "Plant Changes Its Profile"]

[Text] The collective of the industrial association "Pavlograd Chemical Plant" is engaged in production for the public. Production of goods for widespread consumption

has been set up quickly in shops in which only orders from defense departments were placed until recently.

D. Kachalov, chief engineer of the association said that the first stages of conversion were difficult. Instead of the system of orders and list of articles used for 10 years, we were able to select the assortment of goods and the places where they may be sold. The plant's business immediately "fell off" to an amount of 100 million rubles and the collective faced the task of filling the gap in a very short time.

The enterprise collective plans to contact foreign firms. The currency earned will be used to purchase the newest technology and equipment.

### Wrong Nitrogen - Phosgene Escape

907M0148C Moscow *RABOCHAYA TRIBUNA*  
in Russian No 40, 13 Feb 1990 p 1

[Article by S. Sadoshenko: "Nitrogen" But Not Right Kind]

[Text] A poisonous war gas with suffocating action, phosgene, was produced, until very recently, at an enterprise producing articles for purely peaceful purposes, very near the center of Dneprodzerzhinsk.

The characteristics of this gas may be found in any civil defense textbook but Dneprodzerzhinsk townspeople do not need textbooks for they have experienced the effect of phosgene more than once. The industrial association "Azot" recorded more than 46 outbursts of gas last year and more than 60 outbursts occurred in 1988.

An outburst which occurred on 6 November 1989 was almost beyond endurance, said M. Shlenchak, Dneprodzerzhinsk procurator. Twenty workers in neighboring plants were poisoned and hospitalized when the wind carried a phosgene cloud in their direction. Fortunately none of the workers died. The townspeople protested in a demonstration the following day. We, of course, introduced a criminal case immediately. Investigation is now underway.

The Dneprodzerzhinsk industrial association "Azot" is the only producer of commercial phosgene in the country. Until recently, this was a secret for everybody but the local residents. While the plant operated with the newest equipment 10 years ago and was not a hazard to persons wishing to settle there, in recent years the people called more and more resolutely for a halt to production of a "war" gas at a civilian enterprise.

We had already decided to close the shop in 1985, says A. Radchenko, chief physician of the municipal sanitation station. However, the plant managers patched up the equipment and promised that it would work like clockwork but they did not keep their promise. At this point, the townspeople lost their patience. After stormy meetings, the municipal executive committee met twice. A decree was adopted - stop phosgene production until the threat of dangerous discharges of the gas into the atmosphere is completely eliminated. We, with representatives of the procurator, sealed the plant on January 1.

Then another problem arose; what to do about medicinal preparations? You know phosgene is needed to produce some medicines and some consumer goods ranging from linoleum to footwear.

New equipment for the plant has been ordered, says director general of the industrial association "Azot" A. Levchenko. It is to be delivered in the second half of the year. If the Mintyazhmash enterprise, with which we made direct contracts, came near fulfilling this schedule, we would be able to put the shop into operation within a month.

How many years were wasted in the former USSR Ministry of Fertilizers in trying to find a way to prevent use of the equipment of the hazardous plant. Only now when the heat is on and production has been stopped did they begin to become anxious.

What can be done so that there will be synthonycin while ensuring that Dneprodzerzhinsk residents' lives are not in danger. Certainly, "Agrokhim" must provide for construction of a new phosgene shop in a sparsely populated place. It is still necessary to try to convince the residents that when new equipment is installed in "Azot", there will be no more emissions but the shop itself will continue to operate until a new one is built.



UDC 541.182.3

### Aerosols of Laser Plasma

907M0173A Moscow KOLLOIDNYY ZHURNAL  
in Russian Vol 52 No 1, Jan-Feb 90 (manuscript  
received 29 Jan 88) pp 62-66

[Article by A. A. Lushnikov and A. V. Pakhomov,  
Scientific Research Institute of Physical Chemistry  
imeni L. Ya. Karpov, Moscow]

[Abstract] An experimental study was made of solid aerosol formed by the condensation of metal vapor following vaporization of solid metal by a laser beam in an inert atmosphere. Four metals were selected for the study: Ti, Fe, Ag, and Pt. Target specimens of these metals were treated with a Nd-laser operating free and emitting pulses of 1-ms duration or Q-switched and emitting pulses of 20-ns duration, also with a short-pulse CO<sub>2</sub>-laser. They were treated in an He atmosphere and in an Ar atmosphere, and silver was also treated in a hydrogen atmosphere under various pressures below atmospheric. Metal vapor produced by boiling under low pressure was found to condense into spherical particles and formed almost monodisperse aggregates, with their size within the decanometer fraction depending on neither energy nor duration of laser pulses. As the pressure was raised, they condensed into individual particles under intermediate pressures and rapidly into lamellar cluster aggregates under higher pressures. Besides vaporization of metal targets under deep vacuum, flashing those aggregates with optical-breakdown plasma was the only way to produce an aerosol of individual spherical particles by vaporization of metal targets with pulsed laser radiation. Figures 2; tables 3; references 6.

UDC 541.182.2+621.319

### Drift of Aerosol Particles in Strong Nonuniform Oscillating Electric Fields

907M0173B Moscow KOLLOIDNYY ZHURNAL  
in Russian Vol 52 No 1, Jan-Feb 90 (manuscript  
received 2 Feb 88) pp 100-105

[Article by G. I. Surovtsova and V. I. Turubarov, Leningrad Institute of Aviation Instrument Design]

[Abstract] Motion of charged spherical aerosol particles in an alternating electrical field without constant component is treated as an Oseen flow and analyzed accordingly. The corresponding equation is solved by successive approximations, with a Stokes friction and a uniform electric field in the first approximation. In the second approximation the velocity of particles is still a periodic function of time but has two components, one along the lines of force in the electric field and one along the gradient of the field intensity squared. This is then used as the basis for considering the drift of aerosol particles in a medium between two plane-parallel electrodes (capacitor plates), with drift occurring here only

in an asymmetrical external field such as one with a second-harmonic component, in a medium between cylindrical or spherical electrodes (capacitor plates) with possible size segregation of particles on different stabilized equilibrium surfaces within the interelectrode space, and in a medium with a conducting ring between two parallel grounded plates. Figures 4; references 6.

UDC 543.33.284:541.182.644

### Dependence of Silica Gel Structure on Composition of Original Sodium Silicate

907M0173C Moscow KOLLOIDNYY ZHURNAL  
in Russian Vol 52 No 1, Jan-Feb 90 (manuscript  
received 16 Nov 87) pp 112-115

[Article by N. G. Belotserkovskaya, D. P. Dobychin, V. M. Zinyakova, G. M. Kesareva, V. I. Malkima, and V. M. Shamrikov, Leningrad Pedagogical Institute imeni A.I. Gertsen]

[Abstract] An experimental study concerning structuring of silica gel was made, two sets of specimens having been prepared from sodium silicate with moduli of 1.0 and 2.8, respectively. Silica sols were produced by the passage of 3 percent SiO<sub>2</sub> solutions of each grade of sodium silicate through a column of acidic ion-exchange resin, their specific surface then being measured by potentiometric titration and their grain size calculated on the basis of the globule model. They were subsequently gelatinized at room temperature and, while in the form of hydrogels, were heat-treated at 200°C. The absorption characteristics of both sets of silica gel specimens were determined from their benzene vapor and water vapor sorption isotherms. The sorption of these two vapors under pressures covering the range  $p/p_s = (8 \times 10^{-4} \text{ to } 0.98)$  was not only measured at several temperatures with a vacuum adsorption balance but was also calculated on the basis of their true and apparent density. Structural parameters, including skeleton and pore dimensions, calculated by applicable formulas indicate that silica gel produced from sodium silicate with the smaller modulus has smaller pores and thus is more compact, while silica gel produced from sodium silicate with the higher modulus contains branched polymer compounds in a much looser packing. Figures 1; tables 2; references 19.

UDC 535.32:541.182.42

### Refractometric Method of Studying Structural Characteristics of Perfluorocarbon Emulsion Particles

907M0173D Moscow KOLLOIDNYY ZHURNAL  
in Russian Vol 52 No 1, Jan-Feb 90 (manuscript  
received 24 Feb 88) pp 132-135

[Article by I. N. Kuznetsov, Leningrad Scientific Research Institute of Hematology and Blood Transfusion, and A. G. Bezrukova, Leningrad Polytechnic Institute]

[Abstract] Determination of the structural characteristics of finely dispersed perfluorocarbon emulsion particles by the refractometric method is considered, and an experiment is conducted with emulsions in perfluorotributylamine and in perfluorodecalin with perfluorotripropylamine in a 7:3 ratio. These emulsions were prepared by homogenization under high pressure in an aqueous solution of the proxanol-268 surfactant with or without yolk phospholipids. The average diameter of the emulsion particles was measured with a spectroturbidometer, and the refractive index of these two-layer media was measured with an Abbe refractometer. An evaluation of the data and a theoretical analysis based on the model of a sphere inside a concentric spherical shell reveal the dependence of the refractive index on the volume fraction of the dispersed phase and thus also on the relative thickness of the shell, which decreases appreciably even when the latter decreases slightly. Dilution with water was found not to change the refractive index of stable emulsions but to decrease the refractive index of unstable ones, a thin adsorption layer playing an important role here. The results validate the rule that the overall refractive index is equal to the volume-weighted sum of those of the two phases. Figures 4; references 17.

UDC 532.135:541.182.6:541.67

#### Effect of Electric Field on Rheological Characteristics of $\text{TiO}_2$ Suspensions in Solutions of Cellulose Ethers

907M0173E Moscow KOLLOIDNYY ZHURNAL  
in Russian Vol 52 No 1, Jan-Feb 90 (manuscript  
received 23 Feb 88) pp 141-144

[Article by T. G. Lazareva, Ye. V. Korobko, I. N. Yermolenko, and Z. P. Shulman, Institute of Heat and Mass Transfer imeni A. V. Lykov, Institute of General and Inorganic Chemistry, BSSR Academy of Sciences, Minsk]

[Abstract] An experimental study of  $\text{TiO}_2$  suspensions in mixed cellulose acetate ethers was made, its object being to determine the effect of an electric field on their viscosity and on the hydrogen bonds in such a system. Two ethers were selected, acetyl cellulose and cellulose acetophthalate. Both were in a dioxane solution. Measurements were made with a rotary electroviscosimeter, with a specimen filling the gap between two coaxial cylinders and a constant voltage or a 50-Hz alternating voltage from standard sources or an alternating voltage from a special variable-frequency (3-1000 Hz) source applied across them. The electrorheological effect, namely, the relative change in viscosity as a function of the electric field intensity, was calculated from the data. The results indicate that the hydrogen bonds in a "pure" ether solution are not affected by a constant electric field or a 10-300 Hz alternating one of 0.5-3.0 kV/mm intensity, but the presence of the inorganic additive in concentrations even as low as 2-5 percent by weight makes the system sensitive to an electric field. The magnitude of the electrorheological effect was found to depend on the electric

field intensity, increasing monotonically and fast in a constant electric field as the intensity of the latter is raised and saturating fast in an alternating electric field as the frequency of the latter is raised. Its dependence on the  $\text{TiO}_2$  concentration, which was different in a constant electric field and in an alternating one, follows different trends for each kind of ether, just as its rate of rise in a constant electric field and its saturation level in an alternating one is different for each kind of ether. The electrorheological effect was found to become maximum at a different  $\text{TiO}_2$  concentration in each kind of ether solution, this concentration as well as the magnitude of this maximum effect also depending on the strain rate and thus on the response of the system components to the electric field. Measurements made after the voltage had been turned off have yielded the relaxation characteristics, specifically the dependence of the viscosity relaxation time on the voltage and on the strain rate, indicators of thixotropic recovery. Figures 5; references 9.

UDC 541.182.65.047

#### Heteroadagulation of Gold Hydrosol on Inorganic Adsorbents

907M0173G Moscow KOLLOIDNYY ZHURNAL  
in Russian Vol 52 No 1, Jan-Feb 90 (manuscript  
received 15 Feb 88) pp 158-162

[Article by N. V. Nikolenko and Ye. A. Nechayev (deceased), Institute of Chemical Technology imeni F. E. Dzerzhinskiy, Dnepropetrovsk]

[Abstract] Heteroadagulation of gold hydrosol on inorganic adsorbents is described and analyzed by using the concept of resonance ionization potential corresponding to maximum adsorption and an analogy with chemisorption of an organic substance by inorganic sorbents. The equation for heteroadagulation kinetics, combined with the Smolukhovskiy equation for the coagulation rate, yields an equation for the adagulation rate with a trinomial on the right-hand side and a coefficient in each term. This equation is simplified for calculation of two of the coefficients on the basis of the characteristic heteroadagulation time, assuming zero coverage of the adsorbent surface and a zero area fraction covered by sedimentation of sol particles after their coagulation during a short initial period of time and especially when the ratio of metal surface area to adsorbent surface area is small. The third coefficient is calculated by simultaneous solution of the original equation and an equation describing the dependence of the adsorbent surface coverage on the hydrosol molarity, the latter equation being obtained from the rate of surface coverage. All three coefficients, heteroadagulation rate constants, have been evaluated theoretically by numerical solution of those two equations as well as experimentally for gold hydrosol with 0.20 and 0.27 ratios of metal surface area to adsorbent surface area. They are now correlated with the resonance ionization potentials of 17 adsorbent substances: Si,  $\text{SiO}_2$ , quartz,  $\text{TiO}_2$ , CuO, ZnO,  $\text{SnO}_2$ ,  $\text{PbO}_2$ ,



$\text{Bi}_2\text{O}_3$ ,  $\text{ThO}_2$ ,  $\alpha\text{-Al}_2\text{O}_3$ ,  $\gamma\text{-Al}_2\text{O}_3$ ,  $\alpha\text{-Fe}_2\text{O}_3$ ,  $\text{Co}_2\text{O}_3$ ,  $\text{Cr}(\text{OH})_3$ ,  $\text{TiN}$ , and  $\text{WC}$ ). According to the correlation, colloidal gold particles interact most intensely with substances having a resonance ionization potential in the vicinity of 7.25 eV, 7.8 eV, 9.5 eV, and 10.3 eV. For a correlation with the electronic structure of the gold surface, direct adsorption of several organic substances by particles of blue gold hydrosol was measured and the readings compared with the resonance ionization potentials of those substances. Figures 1; tables 1; references 7.

UDC 541.182.41

#### Size of Particles in Microemulsion Systems

907M0173H Moscow KOLLOIDNYY ZHURNAL  
in Russian Vol 52 No 1, Jan-Feb 90 (manuscript  
received 21 Jan 88) pp 162-166

[Article by A. V. Sineva, V. N. Matveyenko, and Ye. D. Shchukin, Department of Chemistry, Moscow University]

[Abstract] An experimental study of the octane-water-surfactant microemulsion systems by photon correlation spectroscopy was made for the acquisition of reliable data on the size distribution of microdroplets in them. Amyl alcohol as an optically active agent was added to all, and a different surfactant was added to each: dodecyl sodium sulfate, cetyl pyridine bromide, OP-10, TRS, or petroleum sulfonate, their concentration being varied as well as the octane: water ratio. Spectroscopy of the system with dodecyl sodium sulfate has yielded the most information about the size distribution of microdroplets, namely, the dependence of their average radius and size-mass distribution on both the surfactant concentration (percent by weight) and the octane:water volume ratio, as well the most information about the dependence of octane and water solubilization and the kinematic viscosity on that ratio. The data indicate that two kinds of particles exist in these emulsions, with the smaller ones being octane solubilizers and the larger ones being water solubilizers so that predominance of one or the other kind depends on the octane:water volume ratio. Figures 4; references 19.

UDC 531.2:537.217:637.127.1

#### Aureole Formation Around Milk Fat Globules

907M0173I Moscow KOLLOIDNYY ZHURNAL  
in Russian Vol 52 No 1, Jan-Feb 90 (manuscript  
received 18 Nov 87) pp 167-168

[Article by I. I. Strakulenko, Moldavian Scientific Research Institute of Irrigation Farming and Vegetable Growing, Tiraspol]

[Abstract] An experimental study of natural milk with a polydisperse 0.1- to 10- $\mu\text{m}$  distribution of particles was made in search of aureole formation around its fat globules and for an explanation of this phenomenon. Specimens of milk were examined under an MBI-6

microscope with a 7x eyepiece and either 40x or 90x objective, taking into consideration the colloidal characteristics of milk and the complex structure of milk fat. In milk diluted with whey in a 2:1 ratio, aureoles began to form around larger fat globules only after 10-15 minutes. In milk further diluted to a 3:1 ratio, smaller fat globules were drifting away from larger ones in a Brownian motion, evidently by the action of electromagnetic repulsion forces, until they stopped at a certain distance but continued to oscillate with a 0.5- to 0.8- $\mu\text{m}$  amplitude. The coming to a standstill is attributed to attraction forces in the system that oppose and eventually balance the repulsion forces. No aureole formation was detected in milk diluted with water distillate 2:1, but aggregation was as a consequence of a lower salt concentration in the disperse medium and fixed positions of fat globules relative to one another. In view of the biological nature of milk fat globules, it is possible that electromagnetic forces in this disperse medium are generated by coherent vibrations at a frequency on the order of  $10^{11}$  to  $10^{12}$   $\text{s}^{-1}$ . Figures 1; references 8.

UDC 541.182.046:546:546.3-14

#### Stability of Liquid-Metal Colloids

907M0173J Moscow KOLLOIDNYY ZHURNAL  
in Russian Vol 52 No 1, Jan-Feb 90 (manuscript  
received 16 Mar 88) pp 175-178

[Article by A. I. Fedonenko, Kharkov University]

[Abstract] An experimental study of magnetic colloids dispersed in liquid metal was made to determine their sedimentation stability on the basis of their easily measurable magnetic susceptibility as an indicator of their composition and dispersion. Iron particles of the 4-nm size fraction were dispersed in mercury, the ferromagnetic content in the system being 2.16 vol. percent in one specimen and 2.68 vol. percent in another. Diffusional growth of these particles during aging was prevented by the addition of antimony or tin, which formed monolayers on their surface. Measurements were made with the colloidal fluid in 11-cm-long vertical tubes 0.7 cm in diameter onto which freely movable 0.4-cm-long inductance coils were slipped. The sedimentation rate served as an indicator of the changing vertical profile of particle concentration. A direct current of 0.2 A was passed through colloidal fluid in one tube by the application of a voltage to it through a pair of electrodes. During a 72-120 hour long initial period the sedimentation rate was approximately the same in both tubes, but afterwards the sedimentation rate was higher in the tube without electrodes. Reversal of the voltage polarity did not change the sedimentation rate in the other tube, which indicates that electrophoresis plays no role in the sedimentation process. Rather, as compact colloidal clusters with lower magnetic susceptibility form and the interlayers separating them become thinner during the stratification process in an electric field, a repulsion force builds up and forms a positive barrier that impedes further thinning of the interlayers so that coagulation of

particles slows down. The origin of this repulsion force can be traced to weakening of the interatomic interaction and to the formation of intermetallic compounds, large changes in the electronic component of the disjoining pressure being ultimately responsible for scattering of electrons at the interphase boundaries and by the inhibitor metal (Sb or Sn) inclusions that stabilizes the sedimentation rate. These conclusions about the sedimentation stabilizing mechanism are validated by the results of rheological measurements in a viscosimeter with a flat channel, using passive Sn rather than active Sb as stabilizer, and with the voltage varied so as to reveal the current dependence of the flow stress characteristic of thixotropic Bingham fluids. Figures 5; references 11.

UDC 541.183.022+661.185.1:532.3[635+64]

#### Kinetics of Surfactant Monolayers Spreading over Water Surface

907M0173K Moscow KOLLOIDNYY ZHURNAL  
in Russian Vol 52 No 1, Jan-Feb 90 (manuscript  
received 25 Nov 87) pp 183-185

[Article by V. G. Chalenko, A. V. Arkharov, L. V. Glubokova, and A. N. Leontyeva, Kiev University]

[Abstract] Spreading of monolayers of higher fatty alcohols over the water surface was studied in an experiment involving measurement of the spreading speed, contamination of the monolayer in the process being prevented by diverting it with a transverse water stream all the time during which its front was approaching a wall of the tray. Preliminary tests revealed a slow transverse flow of the entire water surface layer even in the absence of a surfactant, thus indicating the existence of a hydrodynamic pressure head. Lycopodium granules floating on the surface served as flow tracers, first without and then with 1-dodecanol added as a surfactant. The velocity of tracer granules sticking to the layer edge and moving with it was determined on the basis of distance and time readings. The distance traveled was found to be a power function of time  $s = kt^n$ , and the velocity was accordingly found to be  $v = at^{n-1}$  or  $v = k^{1/n}ns$ , with both  $k$  and  $n$  being constant and thus indicating steadiness. An evaluation of the experimental data on this basis has yielded  $s = 14.8t^{0.82}$  and  $v = 21.9s^{-0.22}$ . Figures 3; references 5.

UDC 541.18.052:542.87:546.821

#### Colloid-Chemical Transformations of Ti-IV During Electrolysis of Aqueous $TiCl_4$ Solutions

907M0173L Moscow KOLLOIDNYY ZHURNAL  
in Russian Vol 52 No 1, Jan-Feb 90 (manuscript  
received 7 Dec 87) pp 188-191

[Article by L. M. Sharygin, S. M. Volk, and L. N. Garidulich]

[Abstract] The formation of  $TiO_2 \cdot nH_2O$  sol during electrolysis of an aqueous  $TiCl_4$  solution was studied by Raman scattering spectroscopy, 5 mole percent  $ZrOCl_2$  having been added to the 1 mol/dm<sup>3</sup>  $TiCl_4$  solution in an electrolyzer without a diaphragm between the titanium cathode and the platinum anode. Samples with successive atomic Ti:Cl ratios were taken periodically during electrolysis with a cathodic current density of 0.2 A/cm<sup>2</sup> at a temperature within 35-45°C. The spectra revealed  $[TiOP]^{-2}$  and  $[Ti_nO_{np}]^{-nz}$  anions along with  $Cl^-$  anions in complexes, with the titanate anions being neutralized by hydrogen cations into titanate acids. They also reveal a shift from a rutile structure to ordered chains and layers of  $TiO_6$  octahedrons growing into colloidal anatase particles. Figures 2; references 13.

UDC 539.217+539.23+541.183.022

#### Permeability of Enzyme Monolayers

907M0173M Moscow KOLLOIDNYY ZHURNAL  
in Russian Vol 52 No 1, Jan 90 (manuscript received 7  
Dec 87) pp 192-196

[Article by T. Yu. Shirokova, N. G. Volkova, Ye. P. Shvyrkov, and A. A. Trapeznikov, Yaroslavl Institute of Medicine, and Institute of Physical Chemistry, USSR Academy of Sciences, Moscow]

[Abstract] The effects of surfactants on the behavior of enzyme monolayers was studied by rheological methods. Solutions of lysozyme, catalase, and crystalline trypsin were selected for the study. Pure dimedrol was used as a surfactant, with NaCl solution in deionized water as the underlayer: 1 mol·m<sup>3</sup> for catalase and trypsin monolayers, 2 mol/dm<sup>3</sup> for lysozyme monolayers. Each enzyme had been tested for homogeneity by the gel filtration method. The salt had been calcined for 4 hours at 100°C and thus cleansed of organic impurities, its solutions then being purified by means of activated carbon. The surface tension at various dimedrol concentrations covering the  $5.2 \times 10^{-5}$  to  $2.6 \times 10^{-3}$  mol/dm<sup>3</sup> range was measured at a constant temperature of 293-294 K by the Wilhelm and Trapeznikov methods using a surface balance, and for determination of its dependence on the specific surface over the 1.2-0.25 m<sup>2</sup> range, the superficial viscosity of monolayers and their modulus of elasticity in shear were measured by the Trapeznikov method using a vibratory system. These measurements indicated the dependence of the monolayer compressibility on the specific surface (with the compressibility dipping to a minimum but less so as more surfactant is added) and the dependence of the viscosity and the shear modulus on the specific surface. The data yield information about the permeability of such pharmaceutical substances that needs to be taken into account in the prescription of antihistamines, narcotics, and other highly surface-active drugs. Figures 3; references 13.

UDC 541.135.52

**Distribution of Potential on Moving Bipolar Electrode**

907M0105A Kiev UKRAINSKIY KNIMICHESKIY  
ZHURNAL in Russian Vol 55 No 12, Dec 89  
(Manuscript received 6 Dec 88) pp 1285-1287

[Article by N. D. Ivanova, T. S. Tsareva, Ye. I. Boldyrev, and O. B. Khrapov, Institute of General and Inorganic Chemistry, Ukrainian Academy of Sciences, Kiev]

[Abstract] A method is suggested for applying coatings to a moving electrode in an electrolyzer in which the moving electrode is bipolar and the current is applied by process electrodes. The use of the bipolar method allows high current density without greatly heating the electrode, thus allowing an increase in the rate of precipitation and thus decreasing the required bath length. The potential of the bipolar electrode decreases with increasing length. The distribution of the potential depends on the position of the positively charged process electrodes. By using the proper distribution of electric field force lines of the positively charged electrodes, it becomes possible to control the distribution of the metal precipitated over the length of the bipolar electrode while maintaining virtually identical potential of the electrode. This allows the production of coatings of virtually identical length over any section of a moving electrode. Figures 4; References 4: Russian.

UDC 541.135:546.78:535.417

**Interferometric Monitoring of Precipitation and Optical Properties of Electrochemical Films of Amorphous WO<sub>3</sub>**

907M0105B Kiev UKRAINSKIY KNIMICHESKIY  
ZHURNAL in Russian Vol 55 No 12, Dec 89  
(Manuscript received 26 Jul 88) pp 1287-1292

[Article by Yu. S. Krasnov, F. N. Patsyuk, and O. A. Sych, Institute of General and Inorganic Chemistry, Ukrainian Academy of Sciences, Kiev]

[Abstract] Results are presented from a study of the process of electroprecipitation of  $\alpha$ -WO<sub>3</sub> by using a computer to control the course of the precipitation and record and analyze interferograms. The use of a computer increases the accuracy of measurement of precipitation parameters and also simplifies determination of the time variation of intensity of the interfering beams. The improved method of interferometric monitoring can

determine the instantaneous values of the parameters of the process and of the optical characteristics of the electrochemical material, including their variation with E. It is found that precipitation in this mode yields a decrease in V with time, but regardless of thickness the growing film has a mirror smooth surface. Figures 2; References 10: Russian.

UDC 541.135

**Coulostatic Method and Consideration of Electrolyte Resistance**

907M0105C Kiev UKRAINSKIY KNIMICHESKIY  
ZHURNAL in Russian Vol 55 No 12, Dec 89  
(Manuscript received 20 Dec 88) pp 1292-1295

[Article by V. I. Chernenko and L. S. Tikhaya, Dnepropetrovsk Institute of Chemical Technology]

[Abstract] Results are presented from an analysis of the influence of electrolyte resistance on coulostatic excitation of an electrode. The coulostatic technique with a discharging condenser requires, in contrast to popular opinion, that the resistance of the electrolyte be eliminated or considered, particularly in studies of rapid electrode processes in weakly conductive media. Figures 3; References 6: 3 Russian, 3 Western.

UDC 621.382

**Photosensitivity of GaAs-Electrolyte Interface With Strong Light Absorption**

907M0105D Kiev UKRAINSKIY KNIMICHESKIY  
ZHURNAL in Russian Vol 55 No 12, Dec 89  
(Manuscript received 6 Jan 89) pp 1295-1298

[Article by N. D. Dmitruk, G. Ya. Kolbasov, and N. I. Taranenko, Institute of General and Inorganic Chemistry, Ukrainian Academy of Sciences, Kiev]

[Abstract] A study is made of the influence of chemical modification of the surface and gamma radiation on the steady photosensitivity of an n-GaAs electrode in the ultraviolet area of the spectrum and the relaxation kinetics of the photo-emf with pulsed UV excitation. It is found that  $J_p$  increases most strongly after modification of the surface by rhenium and decreases after gamma-irradiation of the electrode, which causes both surface and volume defects in the semiconductor and the corresponding recombination centers. Figures 3; References 6: Russian.



UDC 538.1;539.213;541.1

**High-Temperature Superconductivity in  $\text{La}_{2-x}\text{Sr}_x\text{NiO}_4$  System**907M0104A Moscow ZHURNAL  
NEORGANICHESKOY KHIMII in Russian Vol 34  
No 12, Dec 89 (Manuscript received 30 May 89) pp  
3201-3203

[Article by V. A. Ivanov, Institute of General and Inorganic Chemistry imeni N. S. Kurnakov, USSR Academy of Sciences]

[Abstract] It is assumed that in compounds in the system  $\text{La}_{2-x}\text{Sr}_x\text{NiO}_4$  the close approach of  $\text{NiO}_2$  layers decreases the tetragonal component of the crystalline field and  $e_g^2$ -degeneration of the  $3d(x^2-y^2)$  and  $3d(3z^2-z^2)$  orbitals. Single-frequency d-excitations are formed as a result of collectivization of transitions between the basic triplet and the polar three-particle states. The kinematic high-temperature superconductivity mechanism predicts high-temperature superconductivity in compounds with degenerate d electrons such as  $\text{La}_{2-x}\text{Sr}_x\text{NiO}_4$ . References 6: 4 Russian, 2 Western.

UDC 666.655

**Study of Phase Transitions in Specimens of  $\text{CaO-TiO}_2\text{-H}_2\text{O}$  System Obtained by "Sol-Gel" Method**907M0104B Moscow ZHURNAL  
NEORGANICHESKOY KHIMII in Russian Vol 34  
No 12, Dec 89 (Manuscript received 19 Sep 88) pp  
3203-3204

[Article by N. G. Roslyakova, L. Ye. Kademina, G. I. Mamayev, and O. Yu. Sannikova, Leningrad Institute of Technology imeni L'ensovet]

[Abstract] The "sol-gel" method is used to obtain very finely dispersed powders in the system  $\text{CaO-TiO}_2\text{-H}_2\text{O}$ . Phase transitions in the powders and tableted specimens are studied during heat treatment at 60-1,100°C. The specific surfaces of the powders and its variation with heat treatment are determined. Low-temperature phase transitions are discovered. The experimental data indicate that acceleration of chemical reactions causes semidry pressing of the powders to yield calcium metatinate after heating to 700-1,100°C. References 4: Russian.

UDC 541.124-165:546.42'723-31+546.431'723-31

**Synthesis of Solid Solutions and Phase Equilibria in  $\text{BaFe}_{12}\text{O}_{19}\text{-SrFe}_{12}\text{O}_{19}$  System**907M0104C Moscow ZHURNAL  
NEORGANICHESKOY KHIMII in Russian Vol 34  
No 12, Dec 89 (Manuscript received 01 Nov 88) pp  
3205-3206

[Article by Ye. V. Tkachenko and O. V. Bushkova, Urals State University imeni A. M. Gorkiy]

[Abstract] The purpose of this work was to determine the mutual solubility of barium and strontium hexaferrites over a broad range of concentrations and to study the sequence of phase transitions during the synthesis of solid solutions based upon these compounds. Studies were performed by x-ray phase and x-ray structural analysis and construction of curves showing the relationship between composition and the parameters and volume of a unit cell, specific magnetization, coercive force, and melting point. The substances were found to have limited mutual solubility. The synthesis of solid solutions passes through a stage of formation of  $\text{BaFe}_2\text{O}_4$  and the compound  $\text{SrFeO}_{3-x}$ . Figure 1; References 4: Russian.

UDC 546.881'33:541

**Diagram of Phase Relations in  $\text{CdO-MoO}_3\text{-V}_2\text{O}_5$  System**907M0104D Moscow ZHURNAL  
NEORGANICHESKOY KHIMII in Russian Vol 34  
No 12, Dec 89 (Manuscript received 15 Dec 88) pp  
3206-3208

[Article by T. I. Krasnenko and B. V. Slobodin, Institute of Chemistry, Ukrainian Division, Sverdlovsk]

[Abstract] A study is made of the phase composition of the system  $\text{CdO-MoO}_3\text{-V}_2\text{O}_5$  and a diagram of the phase relationships in the subsolidus area is constructed. The formation of a solid solution based on a high temperature modification of cadmium metavanadate  $\text{Cd}_{1-x}\text{V}_{2-2x}\text{Mo}_2\text{O}_6$  is demonstrated. The data contradict the affirmation of Kozlowski, Ziolkowski, et al., that  $\text{MoO}_3$  does not dissolve in  $\text{Cd(VO}_3)_2$ . Figure 1; References 12: 3 Russian, 9 Western.

UDC 546.284-31

**Radiolysis of Modified Silica**907M0113E Moscow ZHURNAL FIZICHESKOY  
KHIMII in Russian Vol 63 No 12, Dec 89 (Manuscript  
received 4 Nov 88) pp 3364-3367

[Article by M. N. Danchevskaya and O. G. Ovchinnikova, Moscow State University imeni M. V. Lomonosov]

[Abstract] A study is presented of the influence of gamma radiation on silica that has been hydrothermally modified in the presence of tetra-N-methylammonium hydroxide and methyl alcohol. The EPR method is used to identify the radiolysis products, and it is determined that graft  $\text{SiOCH}_2$  radicals are formed. For the silica modified with tetra-N-methylammonium hydroxide, the radiolysis products are the graft radicals  $\text{SiOCH}_2$  and  $\text{SiNHCH}_2$ , as well as methyl radicals sorbed by the silica matrix. The formation of  $\text{SiNHCH}_2$  radicals is explained by radiolysis of the N-C bond with subsequent migration of hydrogen from the carbon atom to the nitrogen atom. The formation of the adsorbed  $\text{CH}_3$  radicals results from

the presence in the matrix of graft dimethylamine structures rather than the  $\text{SiOCH}_3$  fragments. Figures 3; References 10: 6 Russian, 4 Western.

UDC 546.18'33'32'171.1

**Properties of Binary Nitrides Synthesized in Na-P-N and K-P-N Systems**

907M0171A Riga IZVESTIYA AKADEMII NAUK LATVIYSKOY SSR: SERIYA KHIMICHESKAYA No 1, Jan-Feb 90 (manuscript received 25 May 89) pp 35-39

[Article by A. A. Vitola, Ya. V. Ronis, and T. N. Miller, Institute of Inorganic Chemistry, LaSSR Academy of Sciences]

[Abstract] Several crystalline binary nitrides have been synthesized:  $\text{NaPN}_2$  (by heating  $\text{NaH}$  with crystalline  $\text{PN}_2\text{H}$  in ammonia at 820-85°C),  $\text{NaP}_4\text{N}_7$  and  $\text{Na}_3\text{P}_6\text{N}_{11}$  rather than  $\text{Na}_2\text{P}_3\text{N}_9$  (by heating  $\text{NaH}$  with  $\text{P}_3\text{N}_5$  or  $\text{PN}_2\text{H}$  in ammonia at 820-850°C), as well as their isostructural crystalline  $\text{KP}_4\text{N}_7$  and  $\text{K}_3\text{P}_6\text{N}_{11}$  analogues. They have all been found to be thermally stable up to 900°C in ammonia and in inert atmospheres. They were found to differ from the standpoint of chemical stability, however, with  $(\text{Na},\text{K})\text{P}_4\text{N}_7$  hardly decomposing at all and  $(\text{Na},\text{K})\text{PN}_2$  decomposing partially at various rates in boiling water as well as in boiling concentrated aqueous acid ( $\text{HCl}$ ,  $\text{HNO}_3$ ) solutions. Noteworthy is that the  $(\text{Na},\text{K})_3\text{P}_6\text{N}_{11}$  nitrides absorb water and simultaneously release metal ions when in contact with such boiling media, thus revealing ion-exchange characteristics and a similarity to zeolites. Cation exchange also occurs during their solid-phase reaction with alkali salts such as  $\text{NaCl}$  or  $\text{KCl}$  at elevated temperatures. With sulfides of divalent metals such  $\text{ZnS}$  or  $\text{FeS}$  in ammonia at 850°C they form metal-phosphorus thionitrides. Lattice parameters (a) and space group periods (d) of all these nitrides and their hydrates were determined on the basis of x-ray diffraction measurements. Figures 1; tables 3; references 5.

UDC 546.171.1'281:533.9

**Highly Dispersed Titanium Nitride-Silicide Powders**

907M0171B Riga IZVESTIYA AKADEMII NAUK LATVIYSKOY SSR: SERIYA KHIMICHESKAYA in Russian No 1, Jan-Feb 90 (manuscript received 2 Mar 89) pp 40-43

[Article by A.P. Orlov and A.B. Letlen, Institute of Inorganic Chemistry, LaSSR Academy of Sciences]

[Abstract] Specimens of finely dispersed titanium nitride-silicide powders were produced experimentally by treating mixtures of titanium and silicon with a stream of hot nitrogen. The original mixtures consisted of PTOM titanium powder with grains up to 40  $\mu\text{m}$  in size and KPS-3 silicon powder with grains in the 20-to-40- $\mu\text{m}$  size fraction. The laboratory reactor included a 70-kW high-frequency oscillator. A comprehensive study

of the specimens was made including phase analysis in a DRON-2 x-ray diffractometer; chemical analysis for total N, Ti, Si content; neutron-activation analysis for oxygen content; and measurement of the specific surface by standard methods as well as differential thermal and thermogravimetric analyses in a Paulik-Paulik-Erdey derivatograph with 240- to 280-mg samples heated in air at a rate of 10°C/min. The powders produced by this technology were found to be black with a specific surface of 11-22  $\text{m}^2/\text{g}$  and accordingly to be of the 0.05- to 0.09- $\mu\text{m}$  grain size fraction. They were found to consist of  $\text{TiN}$ ,  $\text{Ti}_3\text{Si}_3$ ,  $\text{TiSi}_2$ , and free-Si phases, with their weight fractions depending on the Si:Ti weight ratio in the original powder mixture. Increasing the amount of silicon in the original powder from 5 to 54 percent by weight caused the  $\text{TiN}$  content to decrease and the  $\text{Ti}_3\text{Si}_3$  to increase, with the  $\text{TiSi}_2$  phase and free Si appearing at a level of 37 percent by weight or more silicon in the original mixture. Preference was therefore given to producing Ti-Si-N powders from Ti-Si mixtures containing 2.8-15 percent Si by weight. Oxidation of these Ti-Si-N powders in air was found to be exothermic with a sharp peak, oxidation beginning at 520°C (2.8 percent Si by weight) or higher (540°C with 8.1 percent Si by weight in the original mixture) and its rate peaking within the 580-590°C range. Figures 3; tables 1; references 4.

UDC 541.13

**Voltamperometry of Liquid Membranes, Part 7: Reextraction of Pb-II Ions From Dichloroethane Solutions of Dicyclohexano-18-Crown-6 Complexes**

907M0171C Riga IZVESTIYA AKADEMII NAUK LATVIYSKOY SSR: SERIYA KHIMICHESKAYA in Russian No 1, Jan-Feb 90 (manuscript received 24 Feb 89) pp 44-50

[Article by A. N. Popov, T. V. Yevlanova, and S. K. Timofeyeva, Institute of Inorganic Chemistry, LaSSR Academy of Sciences]

[Abstract] An experimental study was made concerning reextraction of Pb-II ions from dichloroethane solutions of dicyclohexano-18-crown-6 +  $\text{Pb}(\text{NO}_3)_2$  complexes. A solution of its A-isomer was obtained by extraction of 0.01 M  $\text{Pb}(\text{NO}_3)_2$  with an equal volume of 0.005 M crown ether solution in dichloroethane. The resulting dichloroethane solution of that complex was subsequently used as a liquid membrane separating two aqueous solutions of magnesium salts, nitrate and acetate, for reextraction and measurement. The effectiveness of such an electrodialysis in terms of the Pb content in both cells was evaluated on the basis of the current-voltage characteristic of that membrane, with the trend of hardly being influenced by  $\text{NO}_3^-$  anions but strongly influenced by the presence of  $\text{CH}_3\text{COO}^-$  anions and



depending on their concentration. Measurements covering the 0-600 V voltage range were therefore made with an ion-selective electrode. Acetate anions shaped the volt-ampere characteristic into a bell curve, with the current peaking at about 300 V to a maximum proportional to the complex concentration in the membrane. A theoretical analysis of this Pb reextraction mechanism, supported by close agreement between measured and calculated volt-ampere curves, confirms its effectiveness. Figures 6; references 8.

UDC 541.49:546.65:547.898

#### **Sorption of Lanthanides by Polymer Sorbent Containing Diaza-18-Crown-6**

907M0171D Riga IZVESTIYA AKADEMII NAUK LATVIYSKOY SSR: SERIYA KHIMICHESKAYA in Russian No 1, Jan-Feb 90 (manuscript received 15 Dec 88) pp 62-66

[Article by S. V. Beltyukova, Ye. V. Malinka, T. B. Kravchenko, A. S. Roska, and A. Kh. Zitsmanis, Institute of Physical Chemistry imeni A. V. Bogatskiy, UkSSR Academy of Sciences, and All-Union Scientific Research Institute of Applied Biochemistry, Scientific-Industrial Association Biolar]

[Abstract] Sorption of thenoyl trifluoroacetates of rare-earth elements in the lanthanide series by a polymer sorbent, namely, styrene-divinylbenzene copolymer with 3.14 mmol/g of the diaza-18-crown-6 macrocyclic polyether, was studied in an experiment after the sorption process had been stabilized and equilibrium conditions reached. The sorbent had been soaked in chloroform for 24 hours, after which the solution was passed through a filter before being dried first between sheets of filter paper and then for 30 minutes in air. Oxides of lanthanides from La to Lu were dissolved in HCl, whereupon the solutions were left to evaporate until wet solid salts remained, and these were then diluted with water to a 0.1 mol/dm<sup>3</sup> concentration. The metal content was determined by complexometric titration with the arsenaza-I reagent. The hydrogen-ion activity was measured with a pH-340 meter, and the sorption was measured by the luminescence method, with luminescence of Eu or another element being excited by light coming from a mercury-quartz lamp through a filter. The most effectively sorbed ions were those of Eu, Sm, and Gd, with the Eu detection threshold being 0.00005 µg/ml. The least effectively sorbed ions were those of Ce, Pr, Yb, and Lu. The intensity of luminescence was found to depend on the sorption time, the pH of the solution, and the trifluoroacetate concentration in it, with the dependence on each of these factors being characterized by a peak. In the case of europium, sorption was completed within 15-30 minutes, with maximum intensity from a neutral solution and very slowly from solutions with a pH on both sides of the 6.0-8.0 range. Various

solvents with different donor-acceptor properties were added in amounts equivalent to 20 vol. percent (water, dimethyl sulfoxide, dimethyl formamide, methanol, ethanol, propanol, acetone). They were found to lower the intensity of luminescence, a solvent with a higher dielectric constant causing it to decrease more. An exception to this pattern was acetone, probably because it forms chemical bonds with donor groups. The addition of various surface-active agents (cetyl-trimethylammonium bromide, tetramethylammonium bromide, cetylpyridine bromide, tetrabutylammonium bromide) was also found to abate sorption of europium to the extent of inhibiting its completion. Figures 4; tables 2; references 13.

UDC 541.182.644:678.744.72

#### **Gel Formation in Polyvinyl Alcohol Containing Boric Acid**

907M0173F Moscow KOLLOIDNYY ZHURNAL in Russian Vol 52 No 1, Jan-Feb 90 (manuscript received 28 Dec 87) pp 154-158

[Article by L. I. Moneva and I. T. Mladenov, Higher Institute of Chemical Technology, Sofia, Bulgaria]

[Abstract] Gel formation in polyvinyl alcohol (PVA) solutions containing boric acid during titration with alkali hydroxides was studied experimentally on two kinds of alcohol specimens with the following respective degrees of polymerization and residual acetate groups: (1) 1120 and 6.9 percent and (2) 1020 and 1.5 percent. They were reprecipitated from aqueous solution by a 1:1 methanol-acetone mixture before being dissolved with stirring for 2 hours at 363 K and then neutralized at 333 K. Their viscosity was measured with a Hepler rheoviscosimeter and an Oubelode viscosimeter at 293 K. Potentiometric titration with an ionic strength of 0.10 was performed with a Radelkis OP-211/1 digital laboratory pH-meter by using glass and calomel electrodes at a temperature of 293 K. It was also performed at a temperature of 293 K in a nitrogen atmosphere. Films of bimolecular solutions were poured onto a slide for replication by using a carbon-platinum composite and examination under an electron microscope with appropriate magnification. Measurements made at temperatures covering the 293-333 K range reveal that the viscosity of neutralized bimolar PVA:H<sub>3</sub>BO<sub>3</sub> solutions decreases monotonically and fast with rising temperature, this process reversing as the temperature drops back. Evidently cyclic complexes formed by boric acid and the presence of lateral acetate groups make the polymer chains less flexible and thus cause the characteristic viscosity of the PVA solutions to increase as the molar H<sub>3</sub>BO<sub>3</sub>:PVA ratio is increased beyond 0.05, this ratio corresponding to minimum viscosity before those cyclic complexes have ceased to be soluble. Figures 5, references 16.

UDC 539.186

**Shape of Spectral Lines of Relativistic Atom in Non-resonance Laser**

907M0218A Leningrad VESTNIK  
LENINGRADSKOGO UNIVERSITETA: FIZIKA,  
KHIMIYA in Russian No 1, Feb 1990 Seriya 4  
pp 85-88

[Article by A. V. Nefedov]

[Abstract] The method of Gell-Mann and Low adiabatic formalism was used to study the shape of spectral lines of a relativistic atom found in a non-resonance-laser field. The study produced an expression for probability of a single-photon transition with consideration of radiation corrections and the effect of an external field. References 5: 4 Russian; 1 Western.

UDC 517.946:534.222

**Waveguide Propagation of Slightly Nonlinear Beams in Heterogeneous Medium**

907M0218B Leningrad VESTNIK  
LENINGRADSKOGO UNIVERSITETA: FIZIKA,  
KHIMIYA in Russian No 1, Feb 1990 Seriya 4  
pp 88-91

[Article by M. A. Bisyarin]

[Abstract] Propagation of a slightly nonlinear beam in a heterogeneous medium with different scales of heterogeneity in transverse and longitudinal directions was described and discussed. A formula for slightly nonlinear modes was derived and the connection between such modes, linear modes in an analogous medium and high-frequency linear modes was established. Transverse heterogeneity resulted in much faster subsidence of the wave field upon removal from the axis of the beam than is the case in an homogeneous medium. References 6 (Russian).

UDC 538.214

**Temperature Dependence of Magnetic Susceptibility of Highly Magnetic Inclusions in Silicon and Germanium**

907M0218C Leningrad VESTNIK  
LENINGRADSKOGO UNIVERSITETA: FIZIKA,  
KHIMIYA in Russian No 1, Feb 1990 Seriya 4  
pp 91-92

[T. A. Gevondyan, T. O. Guryeva and L. P. Strakhov]

[Abstract] Temperature dependences of magnetic susceptibility of highly magnetic inclusions isolated by magnetic separation of diamagnetic powders of silicon and germanium were described and discussed. Curie points for highly magnetic inclusions in germanium equalled  $953 \pm 14$  K and  $798 \pm 15$  K. Complex

dependences of magnetic susceptibility on the temperature appeared for magnetically ordered particles in silicon and made it impossible to isolate typical points of phase transitions. References 5 (Russian).

UDC 621.317.321

**Measurement of Surface Photoelectromotive Force in Vacuum by Method of Static Capacitor in Differentiative Regime**

907M0218D Leningrad VESTNIK  
LENINGRADSKOGO UNIVERSITETA: FIZIKA,  
KHIMIYA in Russian No 1, Feb 1990 Seriya 4  
pp 93-95

[Article by B. D. Katunin, K. M. Rashidkhanov and O. A. Trofimov]

[Abstract] An experimental method was proposed and a device for measuring photoelectromotive forces on semiconductor films in a high vacuum or in different gases was described. The study of surface photoelectromotive force was performed on a static measurement capacitor during switching on and off of light of constant intensity. This simplified performance of the experiment and provided a more universal method. The accuracy of determination of the stationary value of the photoelectromotive force may be increased if you consider possible changes of capacitance of the capacitor section under the effect of light. The method can be used on samples of any specific resistance. It is highly sensitive and facilitates performance of experiments in a vacuum or in different gases. The experimental device was illustrated and described. Figures 2; references 11: 4 Russian; 7 Western.

UDC 535.373.2

**Effect of Diffusion Through Crystal-Electrolyte Contact Surface on Structure of Surface Layer of ZnSe Crystals**

907M0218E Leningrad VESTNIK  
LENINGRADSKOGO UNIVERSITETA: FIZIKA,  
KHIMIYA in Russian No 1, Feb 1990 pp 95-97

[R. V. Grigoryev, S. R. Grigoryev and B. V. Novikov]

[Abstract] A study of the effect of Li and Na diffusion through a crystal-electrolyte boundary on the structure of the surface layer and of changes in photoluminescence spectra of the ZnSe crystal associated with this used aqueous solutions of LiOH, NaOH, KOH and HNO<sub>3</sub>. The luminescence intensity of P- and Q-bands of donor-acceptor pairs and exciton-admixture complexes lines depended upon the alkaline ion radius. Figure 1; references 3 (Russian).

UDC 669.018:537.312.62

**Determination of Oxygen Index in  
Superconducting Ceramic  $\text{YBa}_2\text{Cu}_3\text{O}_{6.5+x}$** 

907M0218F Leningrad VESTNIK

LENINGRADSKOGO UNIVERSITETA: FIZIKA,  
KHIMIYA in Russian No 1, Feb 1990 Seriya 4  
pp 101-102[Article by N. P. Bobrysheva, I. A. Zvereva, Ya. S.  
Kamentsev and N. V. Chezina][Abstract] A proposed method of determining the oxygen  
index  $x$  in high-temperature superconducting ceramic

$\text{YBa}_2\text{Cu}_3\text{O}_{6.5+x}$  by determining the number of copper atoms with a formal degree of oxidation of +3 was described and discussed. The value  $x$  was determined by reducing the copper by Mohr's salt and titrating the excess by potassium permanganate. Difficulties associated with the chemical composition of the high-temperature superconducting ceramic showed that all redox processes relate only to the  $\text{Cu}^{\text{III}} + \text{Fe}^{\text{II}}$  yields  $\text{Cu}^{\text{II}}\text{Fe}^{\text{III}}$  transition. Possible non-stoichiometry of the lattice produced by change of the relationship between the metal atoms and corresponding deviations to either side of the 6.5 oxygen index did not affect the value of  $x$ . The value of  $x$  was connected only with the  $\text{Cu}^{3+}$  yields  $\text{Cu}^{2+}$  transition since neither Y nor Ba form heterovalent compounds.

UDC 547.212'147;547.212'162.1

**Synthesis of Diethylzinc From Zinc Ethylate and Triethylaluminum**

907M0195A Moscow METALLOORGANICHESKAYA KHIMIYA in Russian Vol 3 No 2, Mar-Apr 1990 (manuscript received 25 Apr 89) pp 296-298

[Article by N. N. Korneyev, N. N. Govorov, M. V. Tomashevskiy, A. V. Bukhtiarov, I. A. Rodnikov, T. V. Ivanova, and N. N. Khromykh, State Scientific Research Institute of the Chemistry and Technology of Heteroorganic Compounds, Moscow]

[Abstract] An analysis was conducted of the synthesis of diethylzinc by the reaction of zinc ethylate under conditions of a twofold excess of triethylaluminum and equimolar conditions. Gas chromatographic analysis of the products led to the conclusion that equimolar conditions constitute an optimum process for preparative synthesis of diethylzinc. The equimolar process involved a more complete ethyl group exchange according to the following reaction scheme:  $3\text{Zn}(\text{OEt})_2 + 2\text{Et}_3\text{Al}$  becomes  $3\text{ZnEt}_2 + 2\text{Al}(\text{OEt})_3$ . The yield of  $\text{ZnEt}_2$  was on the order of 96 percent. References 10: 5 Russian, 5 Western.

UDC 542.91;541.49;546.98

**Reaction of Organopalladium Compound With Diphenyldiazomethane**

907M0195B Moscow METALLOORGANICHESKAYA KHIMIYA in Russian Vol 3 No 2, Mar-Apr 1990 (manuscript received 6 Apr 89) pp 312-315

[Article by I. N. Busygina, T. A. Stromnova, S. B. Katser, A. S. Antsyshkina, M. A. Poray-Koshits, S. G. Sakharov, and I. I. Moiseyev, Institute of General and Inorganic Chemistry imeni N. S. Kurnakova, USSR Academy of Sciences, Moscow]

[Abstract] Reaction of diphenyldiazomethane (I) with  $\text{Pd}(\text{OAc})_2$  in benzene at 35°C leads to decomposition of I with the elimination of nitrogen (0.5 mole per mole of I) and the formation of benzophenone azine (II) and an organopalladium compound (III.) At temperatures above 60°C in acetic acid-benzene solvent, compound III undergoes transformation into the trinuclear  $\text{Pd}_3(\mu\text{-OAc})_4(\mu\text{-}[\text{NC}(\text{Ph})\text{C}_6\text{H}_4\text{I}_2])$  cluster (IV). The latter complex, in addition to acetate bridges, contains a III bridge with two phenyl rings with Pd atoms in the ortho position. Tabulated data on valence angles and bond lengths for IV derived from x-ray analysis are provided.

Tables 2; references 8: 4 Russian, 4 Western.

UDC 542.97;547.592.3;541.251+541.63

**Zero-Valency Platinum in Redox Troponization of Trichloromethylated Cyclohexadienones**

907M0195C Moscow METALLOORGANICHESKAYA KHIMIYA in Russian Vol 3 No 2, Mar-Apr 1990 (manuscript received 27 Apr 89) pp 330-332

[Article by G. V. Gavrilova, V. A. Nikanorov, V. I. Rozenberg, and O. A. Reutov, Institute of Heteroorganic Compounds imeni A. N. Nesmeyanova, USSR Academy of Sciences, Moscow]

[Abstract]  $\text{Pt}(\text{PPh}_3)_4$  reacts with 4-methyl-4-trichloromethyl-2,5-cyclohexadiene-1-one and its 2-chloro- and methyl derivatives to form the corresponding 4-methyl-5-chloro-2,4,6-cycloheptatriene-1-ones via redox troponization. The reactions were much slower than analogous reactions mediated by  $\text{Pd}(\text{PPh}_3)_4$ , with resultant yields that were 5-30 percent lower after 10 days of reaction. In addition, the  $\text{MCl}_2(\text{PPh}_3)_2$  products obtained with  $\text{Pt}(\text{PPh}_3)_4$  were of the cis- rather than trans-configuration. References 11: 5 Russian, 6 Western.

UDC 541.144.7;541.49;546.735;547.31

**Photochemical Activation of  $\text{Co}_4(\text{CO})_{12}$  for Reaction With Alkynes**

907M0195D Moscow METALLOORGANICHESKAYA KHIMIYA in Russian Vol 3 No 2, Mar-Apr 1990 (manuscript received 6 Jun 89) pp 382-386

[Article by S. P. Tunik, V. R. Krym, and A. B. Nikolskiy, Leningrad State University]

[Abstract] Photoexcitation studies showed that activation of  $\text{Co}_4(\text{CO})_{12}$  at  $<365$  nm, corresponding to low energy absorption bands of the electronic spectrum, led to the formation of quasioctahedral clusters of the  $\text{Co}_4(\text{CO})_{10}\text{RC}_2\text{R}'$  type from alkynes in approximately 82-85 percent yields. This approach was used for the specific synthesis of two novel compounds from propinol and butyndiol that were identified on the basis of chemical analysis and a variety of spectroscopic techniques as  $\text{Co}_4(\text{CO})_{10}(\text{HCCCH}_2\text{OH})$  and  $\text{Co}_4(\text{CO})_{10}(\text{HOCH}_2\text{CCCH}_2\text{OH})$ , respectively. Attempts at excitation of  $\text{Co}_4(\text{CO})_{12}$  with the full spectrum emission of a mercury lamp resulted in a loss of selectivity and the formation of a mixture of products. Figures 2; tables 2; references 8 (Western).

UDC 547.1'13:546.831:678.742.2

**Alkyl-Substituted Zirconocenes in Catalysis of Ethylene Polymerization**

907M0196E Moscow METALLOORGANICHESKAYA  
KHIMIYA in Russian Vol 3 No 2, Mar-Apr 1990  
(manuscript received 11 Nov 89) pp 472-473

[Article by V. P. Maryin, L. A. Vyshinskaya, B. A. Krentsel, and N. I. Ivanova, Scientific Research Institute of Chemistry, Gorkiy State University; Institute of Petrochemical Synthesis imeni A. V. Topchiyev, USSR Academy of Sciences, Moscow]

[Abstract] Alkyl-substituted zirconocenes were tested for their efficiency in catalyzing ethylene polymerization (70°C, 7.5 atm, toluene), and this led to the observation that the isopropyl congener was the most efficient. The efficiency of (iso-PrC<sub>3</sub>H<sub>7</sub>)<sub>2</sub>ZrCl (1.1 x 10<sup>-3</sup> mmol/l Zr, 61.5 mmol/l Al) was attributed to mobile hydride centers on the Zr atoms. An increase in the number of alkyl moieties led to a reduction in activity due to screening of the Zr atom. References 6: 3 Russian, 3 Western.

UDC 542.91:548:737:541.49

**Simultaneous Disruption of C-C and C-O Triple Bonds in Formation of Unique Heptanuclear Mo-Co Cluster**

907M0195F Moscow METALLOORGANICHESKAYA  
KHIMIYA in Russian Vol 3 No 2, Mar-Apr 1990  
(manuscript received 13 Nov 89) pp 474-475

[Article by A. D. Shaposhnikov, M. V. Drab, A. A. Pasynskiy, I. L. Yermenko, S. Ye. Nefedov, A. I. Yanovskiy, and Yu. T. Struchkov, Institute of General and Inorganic Chemistry imeni N. S. Kurnakov, USSR Academy of Sciences, Moscow; Physicochemical Institute imeni A. V. Bogatskiy, Ukrainian SSR Academy of Sciences, Odessa]

[Abstract] Reaction of the binuclear complexes Co<sub>2</sub>(CO)<sub>8</sub>(μ, Ige<sup>2</sup>-PhC[triple bond]CPh) and Cp<sub>2</sub>Mo<sub>2</sub>(CO)<sub>6</sub> in boiling o-xylene led to the synthesis of Cp<sub>4</sub>Mo<sub>4</sub>(μ<sup>3</sup>-OH)(μ<sup>6</sup>-C)Co<sub>3</sub>(μ-CO)<sub>3</sub>(CO)<sub>3</sub> (I.) The latter heptanuclear heterometallic cluster was obtained in the form of dark brown crystals. X-ray and PMR analyses provided data for a geometric formula of I. References 4: 1 Russian, 3 Western.



UDC 543.244.6-41+(541.49:547.466)

**Solution Kinetics of Calcium Hydroxyapatite in Hydroxyethylidene Diphosphonic Acid**

907M0214A Leningrad ZHURNAL OBSHCHEY  
KHIMII in Russian Vol 60 No 3, Mar 90 (manuscript  
received 14 Apr 88) pp 497-501

[Article by T. A. Matkovskaya and N. M. Dyatlova, All-Union Scientific Research Institute of Chemical Reagents and Highly Purified Chemical Substances, Moscow]

[Abstract] A comparative analysis was conducted on the rate of solution of calcium hydroxyapatite in water and hydroxyethylidene diphosphonic acid, which is a drug base and regulator of calcium metabolism, various concentrations of solutions at 298 K, pH 7.4. In both cases the kinetics were described by the Yerofeyev-Kolmogorov equation, with the solution rate much greater in water than in 0.1 M hydroxyethylidene diphosphonic acid solutions. The solution rate of hydroxyethylidene diphosphonic acid then abruptly increases in comparison to that of pure water. The S-shaped nature of solution plots in hydroxyethylidene diphosphonic acid was attributed to differences in solubility of mono- and polynuclear complexes formed by calcium and hydroxyethylidene diphosphonic acid, with the ratio of the complexes changing with the concentration of calcium hydroxyethylidene diphosphonic acid. At low concentrations of hydroxyethylidene diphosphonic acid poorly-soluble polynuclear complexes predominate, whereas at higher concentrations more soluble calcium:hydroxyethylidene diphosphonic acid = 1:1 and 1:2-type complexes are formed. Figures 2; tables 2; references 16: 5 Russian, 11 Western.

UDC 541.67

**Polarity and Electron Interactions in P<sup>II</sup> Compounds. Phosphine-Substituted 3-Iminophosphines**

907M0214B Leningrad ZHURNAL OBSHCHEY  
KHIMII in Russian Vol 60 No 3, Mar 90 (manuscript  
received 12 Jul 88) pp 502-504

[Article by I. I. Patsanovskiy, Yu. Z. Stepanova, E. A. Ishmayeva, V. D. Romanenko, and L. N. Markovskiy, Kazan State University imeni V. I. Ulyanov-Lenin; Institute of Organic Chemistry, Ukrainian Soviet Socialist Republic Academy of Sciences, Kiev]

[Abstract] Dipole moment calculations were used to study the polarity and electron interactions of N-phosphorylated  $\Lambda^3$ -aminoiminophosphines. The results demonstrated that derivatives of  $\Lambda^3$ -aminoiminophosphines bearing a 2,4,6-tri-tert-butylphenyl group on atom N<sup>II</sup>, where the superscript indicates the coordinate number, represent systems with localized P=N bonds. Accordingly, their stability in solution as well as in the

crystalline state is due to spatial shielding of the multiple bonds by the bulky substituent. Tables 1; references 8 (Russian).

UDC 547.241

**S- and N-Phosphorylated Ketene-S, N-Acetals**

907M0214C Leningrad ZHURNAL OBSHCHEY  
KHIMII in Russian Vol 60 No 3, Mar 90 (manuscript  
received 22 Jul 88) pp 505-508

[Article by Ye. V. Zheltova, S. G. Churusova, V. A. Kozlov, A. F. Grapov and N. N. Melnikov, All-Union Scientific Research Institute of Chemical Plant Protection, Moscow]

[Abstract] Infrared and paramagnetic resonance spectroscopies were used to demonstrate that reaction of dithiophosphoric acids with acylated malonodinitrile led to the synthesis of stable S-phosphorylated ketene-S,N-acetals. Reaction of the latter with dimethylformamide acetal led to the formation of ketene-S,N-acetals rather than pyrimidinones. The reaction evidently involved S->N migration of the thiophosphoryl group and subsequent alkylation of an intermediate enethiol. Tables 1; references 3 (Russian).

UDC 547.1'118'547.446

**Reaction of Cyclic Halogenated Ketones With Trivalent Phosphorus Derivatives**

907M0214D Leningrad ZHURNAL OBSHCHEY  
KHIMII in Russian Vol 60 No 3, Mar 90 (manuscript  
received 26 Jul 88) pp 508-510

[Article by L. N. Grishina, O. Ye. Gayfullina, N. P. Dubinina, V. G. Shaydullina and Ye. V. Bayandina, Institute of Organic and Physical Chemistry imeni A. Ye. Arbuzov, Kazan Branch, USSR Academy of Sciences]

[Abstract] Dialkyl phosphites were found to react with 7,7-dichlorobicyclo[3.2.0]hepta-2-en-6-one at room temperature to yield novel  $\alpha$ -hydroxyphosphonates. The latter reacted with acetic acid in an acylation reaction catalyzed by sulfuric acid to yield previously undescribed 6-acetoxy-7,7-dichlorobicyclo[3.2.0]hepta-2-en-6-ylidialkyl phosphonates. Tables 1; references 4 (Russian).

UDC 547.571+547.241

**Prototropic and Phosphorotropic Migrations in Phosphorylated 2-Azaallyl Systems. Part 6. Reactions of N-Benzyl-4-Nitrobenzimidoyl- and N-(4-Nitrobenzyl)Benzimidoyl Chlorides with Triphenylphosphine and Amidophosphites**

907M0214E Leningrad ZHURNAL OBSHCHEY  
KHIMII in Russian Vol 60 No 3, Mar 90 (manuscript  
received 26 Jul 88) pp 523-527

[Article by P. P. Onysko, T. V. Kim, Ye. I. Kiseleva, V. P. Prokopenko, and A. D. Sinita, Institute of Organic

Chemistry, Ukrainian Soviet Socialist Republic Academy of Sciences, Kiev]

[Abstract] Prototropic and phosphotropic migrations in phosphorylated 2-azaallyl systems were studied in reactions of N-benzyl-4-nitrobenzimidoyl- and N-(4-nitrobenzyl)benzimidoyl chlorides with triphenylphosphine. In both situations an identical mixture of isomeric imines is produced, containing a triphenylphosphonium group on the C<sup>1</sup> and C<sup>3</sup> atoms of the azaallyl triad. These observations demonstrated the occurrence of successive prototropic and phosphotropic 1,3-shifts at 20°C. Reaction of the imidoyl chlorides with triphenylphosphine and amidophosphites yielded unstable, bright blue ilides, indicative of the substantial contribution of resonance structures. References 11: 9 Russian, 2 Western.

UDC 547+541.571:541.122

**Electron Donor and Acceptor Functions of Physiologically Active and Model Compounds. Part 15. Application of Multiplicative Factor Rule to Thermodynamics of Hydrogen Bond Between Difluoronitroacetanilides and Phosphorylated Oximes**

907M0214F Leningrad ZHURNAL OBSHCHEY KHMII in Russian Vol 60 No 3, Mar 90 (manuscript received 15 Aug 88) pp 527-530

[Article by V. Yu. Grigoryev, V. P. Solovyev, O. A. Rayevskiy, A. N. Ivanov, V. B. Sokolov, and I. V. Martynov, Institute of Physiologically Active Substances, USSR Academy of Sciences, Chernogolovka]

[Abstract] Trials were conducted on the applicability of the multiplicative factor rule to assessment of the thermodynamic parameters of hydrogen bond formation between difluoronitroacetanilides and phosphorylated oximes in benzene at 298 K. Excellent agreement was obtained between the theoretical calculations and experimental data obtained by calorimetric titration. These observations demonstrated the applicability of the multiplicative factor rule to calculation of electron donor and acceptor factor of these compounds and to assessment of hydrogen bond thermodynamics. Tables 2; references 15: 12 Russian, 3 Western.

UDC 547.582.3:542.924:547.26'118

**Substituent Steric Effects in Reaction of Phosphorus Dithioacids With Cumene Hydroperoxide**

907M0214G Leningrad ZHURNAL OBSHCHEY KHMII in Russian Vol 60 No 3, Mar 90 (manuscript received 20 Jul 88) pp 530-532

[Article by Ye. N. Cherezova, R. D. Sayakhov, V. I. Galkin, O. A. Cherkasova, N. A. Mukmeneva, and R. A.

Cherkasov, Kazan Institute of Chemical Technology imeni S. M. Kirov; Kazan State University imeni V. I. Ulyanov-Lenin]

[Abstract] Studies on the steric effects in the reaction of dithiophosphoric and dithiophosphonic acids with cumene hydroperoxide demonstrated that these reactions proceed with high stoichiometric coefficients. Further, these reactions were shown to be first order in acid and second order in cumene hydroperoxide. Correlation analyses revealed that the stoichiometric coefficient was directly related to the steric effect of the substituent on the sulfur atom of the thiol and their efficiency as electron acceptors. The rate constants, however, were inversely related to the steric effects of the substituents. Tables 1; references 4 (Russian).

UDC 547.26'118

**Reaction of Potassium Salts of N-Phosphorylated Amides and Thioamides with Methyl Iodide**

907M0214H Leningrad ZHURNAL OBSHCHEY KHMII in Russian Vol 60 No 3, Mar 90 (manuscript received 2 Aug 88) pp 533-537

[Article by N. G. Zabiroy, F. M. Shamsevalyev and K. A. Cherkasov, Kazan State University imeni V. I. Ulyanov-Lenin]

[Abstract] Monitoring of the reactions of N-phosphorylated thiobenzamide derivatives and their potassium salts with methyl iodide demonstrated that alkylation affects the thiocarbonyl group. In the case of N-phosphorylated benzamide derivatives alkylation involves the sulfur or oxygen of P=X groups, where X = S or O, leading to the formation of monophosphazines. Failure to detect nitrogen atom methylation was attributed to its low nucleophilicity and steric inaccessibility. Tables 1; references 3: 2 Russian, 1 Western.

UDC 547.26'118

**Reaction of Tert-Butyl Esters and Amide Esters of P(III) Acids With Phenylisothiocyanate**

907M0214I Leningrad ZHURNAL OBSHCHEY KHMII in Russian Vol 60 No 3, Mar 90 (manuscript received 26 Jul 88) pp 563-566

[Article by T. Kh. Gazizov, Yu. V. Chugunov, and L. K. Salkeyeva, Institute of Organic and Physical Chemistry imeni A. Ye. Arbuzov, Kazan Branch, USSR Academy of Sciences]

[Abstract] An analysis was conducted on the reaction products of tert-butyl esters and amide esters of P(III) acids with phenylisothiocyanate, since such reactions have not been previously reported. The results demonstrated that reactions of this type occur at room temperatures over a course of some ten hours, leading to the formation of the corresponding esters, amide esters, and

amides of phenylthiocarbamoylphosphonic acid. In general, the yields are on the order of 75-80 percent. References 9: 6 Russian, 3 Western.

UDC 547.78+547.468

**Preparation and Properties of Mesomeric Imidazole Phosphonium Ildes and Betaines**

907M0214J Leningrad ZHURNAL OBSHCHEY  
KHIMII in Russian Vol 60 No 3, Mar 90 (manuscript  
received 12 Aug 88) pp 566-574

[Article by V. S. Brovarets, O. B. Smoliy, S. I. Vdovenko and B. S. Drach, Institute of Bioorganic Chemistry, Ukrainian Soviet Socialist Republic Academy of Sciences, Kiev]

[Abstract] A summary is presented of a series of studies on the synthesis and properties of mesomeric imidazole phosphonium ildes and betaines. The latter were prepared by reacting phosphorylated imidoyl chlorides possessing the  $-CCl=NCH_2P^+=$  grouping with sodium cyanate or sodium thiocyanate. Ready alkylation and acylation of the phosphonium ilde and betaine compounds yielded novel imidazole derivatives. Figures 2; tables 5; references 6: 5 Russian, 1 Western.

UDC 547.246:548.1'13

**Kinetics of Combined Thermal Dissociation of Gaseous Trimethylarsine and Trimethylaluminum**

907M0214K Leningrad ZHURNAL OBSHCHEY  
KHIMII in Russian Vol 60 No 3, Mar 90 (manuscript  
received 14 Jul 88) pp 574-578

[Article by V. A. Yablokov, A. V. Dozorov, S. V. Mitrofanova, and B. S. Yavich, Gorkiy Civil Engineering Institute imeni V. P. Chkalov]

[Abstract] A comparative study was conducted on the dissociation kinetics of gaseous  $Me_3Al$  and  $Me_3As$  in isolation and their equimolar mixture over a temperature range of 407-455°C and vapor pressures of 40 to 220 mm Hg. It has previously been established that at room temperature such mixtures give rise to  $Me_3Al \cdot Me_3As$  compounds. The kinetic curves demonstrated that in the case of  $AsMe_3$  the constants for thermal dissociation are identical for pure  $Me_3As$  and for the mixture, and that  $AsMe_3$  is the more thermostable component.  $Me_3As$ , in addition, influences thermal dissociation of  $Me_3Al$ , which tends to form  $(Me_3Al)_2$  dimers. As the temperature increases, the compounds and dimers dissociate and the rate of dissociation assumes first order kinetics for  $Me_3Al$ . Figures 1; tables 1; references 8: 3 Russian, 5 Western.

UDC 547.963.4

**Coagulation of Human Oxyhemoglobin by Phenyl Mercury Reagent in Neutral Phosphate Buffer**

907M0214L Leningrad ZHURNAL OBSHCHEY  
KHIMII in Russian Vol 60 No 3, Mar 90 (manuscript  
received 23 Mar 88) pp 685-691

[Article by V. S. Konyayeva and A. Ye. Myshkin, Institute of Chemical Physics imeni N. N. Semenov, USSR Academy of Sciences, Moscow]

[Abstract] An analysis was conducted on the coagulation of human oxyhemoglobin with phenyl mercury ( $PhHg^+$ ) reagent at 20°C and higher temperatures in pH 7.2 phosphate buffer. The data demonstrated that 12-14 moles of the  $PhHg^+$  cation were required per one mole of the oxyhemoglobin tetramer for efficient coagulation, a figure approximately twice as high as for  $Hg^{2+}$  cation. The data also demonstrated that whereas the phenyl mercury iodide form was incapable of initiating coagulation at 20°C, coagulation was obtained at 40°C. On balance, the results showed that while binding of  $Hg^{2+}$  to a phenyl moiety did not abolish its ability to coagulate oxyhemoglobin, its efficiency as a coagulant was reduced two-fold. Figures 5; references 5: 3 Russian, 2 Western.

UDC 541.49:542.61

**Platinum Carbonyl Compound  $(Ph_3PBz)[Pt(CO)Cl_3]$ -Promoted Deoxygenation of Sulfoxides by Carbon (II) Monoxide**

907M0214M Leningrad ZHURNAL OBSHCHEY  
KHIMII in Russian Vol 60 No 3, Mar 90 (manuscript  
received 24 Aug 89) pp 692-693

[Article by V. Yu. Kukushkin and A. I. Moiseyev, Leningrad State University]

[Abstract] Studies were conducted in an atmosphere of carbon monoxide (CO) (1 atm, 20°C, 5-7 days) which demonstrated that  $R_2SO$  ( $R = Me, Et, Pr, Bz$ ) undergoes deoxygenation to  $R_2S$  in the presence of  $(Ph_3PBz)[Pt(CO)Cl_3]$ . In the absence of  $(Ph_3PBz)[Pt(CO)Cl_3]$  reduction of  $R_2SO$  did not take place. In addition, reaction of  $(Ph_3PBz)[Pt(R_2SO)Cl_3]$  with CO under identical conditions was found to yield  $R_2S$  and  $(Ph_3PBz)[Pt(CO)Cl_3]$ . References 5 (Russian).

UDC 547.245

**Reaction of Trimethylsilyl Esters of Phosphoric Acids With Salts of Inorganic and Organic Acids**

907M0214N Leningrad ZHURNAL OBSHCHEY  
KHIMII in Russian Vol 60 No 3, Mar 90 (manuscript  
received 14 Apr 89) pp 693-694

[Article by L. A. Lazukina and V. P. Kukhar, Institute of Bioorganic Chemistry, Ukrainian Soviet Socialist Republic Academy of Sciences, Kiev]

[Abstract] Bis- and tris-(trimethylsilyl)phosphites, tris-(trimethylsilyl)phosphate, and trimethylsilyl polyphosphate react with alkali metal salts of organic and inorganic acids to form trimethylsilyl derivatives of the corresponding acids. The reactions occur at 150-200°C with product yields ranging from 20 to 95 percent. References 5: 3 Russian, 2 Western.

UDC 547.241

#### Amine and Amide Methylation of Bis(Trimethylsiloxy) Phosphine

907M02140 Leningrad ZHURNAL OBSHCHEY KHMII in Russian Vol 60 No 3, Mar 90 (manuscript received 19 Sep 89) pp 699-701

[Article by A. A. Prishchenko, M. V. Livantsov, D. A. Pisarnitskiy, N. M. Shagi-Mukhametova, and V. S. Petrosyan, Moscow State University imeni M. V. Lomonosov]

[Abstract] Highly reactive chloromethylamines (amides) were used for reaction with bis-(trimethylsiloxy)phosphine at room temperature under equimolar conditions for the synthesis of the following phosphonates: R-dimethylaminomethyl phosphonite, R-diethylaminomethyl phosphonite, R-N-methyl-N-acetylaminomethyl phosphonite, and R-pyrrolidone-2-yl-1-methyl phosphonite, where R = O,O-bis-(trimethylsilyl). Reaction of chloromethylamine with excess bis-(trimethylsiloxy)phosphine provided the following phosphinates: R-(dimethylaminomethyl) phosphinate, R-(diethylaminomethyl) phosphinate, R-(N-methyl-N-acetylaminomethyl) phosphinate, and R-(pyrrolidone-2-yl-1-methyl) phosphinate, where R = O-trimethylsilylbis. References 3: 2 Russian, 1 Western.

UDC 547.341+542.91+547.1'118

#### Silyl Phosphites in Todd-Atherton Reaction

907M0214P Leningrad ZHURNAL OBSHCHEY KHMII in Russian Vol 60 No 3, Mar 90 (manuscript received 3 Mar 89) pp 701-702

[Article by E. Ye. Nifantsev, M. A. Kharshan, and S. A. Lysenko, Moscow State Pedagogical Institute imeni V. I. Lenin]

[Abstract] Efficient phosphorylation of secondary amines with alkylsilyl and disilyl phosphites in carbon tetrachloride at -20°C was achieved with triethylamine. The reaction conditions consisted of dropwise addition of 0.1 M of secondary amine and 0.1 M triethylamine in CCl<sub>4</sub> to 0.1 M phosphite in absolute CCl<sub>4</sub>. References 3 (Western).

UDC 547.341

#### Dichlorocyclopropane Addition to Dialkenyl Phosphinates

907M0214Q Leningrad ZHURNAL OBSHCHEY KHMII in Russian Vol 60 No 3, Mar 90 (manuscript received 4 May 89) pp 702-703

[Article by B. G. Liorber, G. V. Urmancheyeva, and V. A. Pavlov, Kazan Institute of Chemical Technology imeni S. M. Kirov]

[Abstract] 2,2-Dichlorocyclopropyl methyl-2-propenylbutyl phosphinate, 2,2-dichlorocyclopropyl methyl-2-propenylmethyl phosphinate, and 2,2-dichloro-3-methylcyclopropenyl methyl-2-propenylbutyl phosphinate were prepared by triethylbenzylammonium chloride-catalyzed reaction of dichlorocyclopropane with dialkenyl phosphinates. The reaction conditions consisted of olefin:chloroform:50 percent NaOH molar ratio of 1:10:14. References 1 (Russian).

UDC 547.341

#### Phosphorylation of N-Acetylthiourea With Phosphoric Pentachloride

907M0214R Leningrad ZHURNAL OBSHCHEY KHMII in Russian Vol 60 No 3, Mar 90 (manuscript received 11 May 89) pp 706-707

[Article by V. G. Rozinov, M. Yu. Dmitrichenko, G. V. Dolgushin, and V. I. Donskikh, Irkutsk State University]

[Abstract] Reaction of PCl<sub>5</sub> with N-acetylthiourea results in the formation of N<sup>2</sup>-dichlorophosphoryl-N<sup>1</sup>-(1-chloroethylidene) chloroformamidine. Reaction of the latter with HCl yielded N-dichlorophosphoryl-N<sup>2</sup>-(1,1-dichloroethyl) chloroformamidine. References 2 (Russian).

UDC 547.972.1

#### Thiofluorescein Derivatives as Efficient Catalysts for Hydrogen Generation From Aqueous Solutions

907M0214S Leningrad ZHURNAL OBSHCHEY KHMII in Russian Vol 60 No 3, Mar 90 (manuscript received 27 Jun 89) pp 708

[Article by A. V. Yeltsov, A. I. Ponyayev, and V. P. Martynova, Leningrad Technological Institute imeni Lensovet]

[Abstract] Assessment of xanthene dyes demonstrated that thioderivatives function as efficient catalysts for hydrogen generation in the following system: 0.001 M dye, 0.1 M triethanolamine, and finely dispersed platinum stabilized by polyvinylpyrrolidone in 0.1 N NaOH (370 nm excitation). The highest efficiency was exhibited by tetrabromothiofluorescein, which retained its efficiency for 200 h involving 500 cycles of illumination. References 4: 2 Russian, 2 Western.

UDC 546.185



**Binary Triamidophosphite-Haloform System as  
Novel Source of Trihalogenmethanide Ions**

907M0214T Leningrad ZHURNAL OBSHCHEY  
KHIMI in Russian Vol 60 No 3, Mar 90 (manuscript  
received 4 Apr 89) pp 709-710

[Article by G. N. Koydan, A. P. Marchenko, and A. M.  
Pinchuk, Institute of Organic Chemistry, Ukrainian  
Soviet Socialist Republic Academy of Sciences, Kiev]

[Abstract] A series of trimethylsilyltri-halogenmethanes were synthesized by reacting triamidophosphites with a haloform and halogen silane. Bis(trimethylsilyl)di-halogenmethanes were obtained by reacting trimethylsilyltri-halogenmethanes with halogen silane and triami-dophosphites. The preparative advantage of this approach lies in greater thermal stability of iodoform over the carbon tetrachloride method. References 6: 3 Russian, 3 Western.

UDC 541.11

**Thermochemistry of Series of Tricyclopentadienyl Compounds of Uranium**

907M0118A Leningrad RADIOKHIMIYA in Russian  
Vol 31 No 6, Nov-Dec 89 (manuscript received  
2 Jan 89) pp 38-40

[Article by V. I. Telnov, I. B. Rabinovich, V. N. Larina,  
M. R. Leonov, and G. V. Solovyeva]

[Abstract] In a previous work the enthalpy of combustion of tetracyclopentadienyl uranium was determined experimentally and this value was then used to calculate its enthalpy of formation and the mean rupture enthalpy of the U-C<sub>5</sub>H<sub>5</sub> bond (equal to 247 +/- 4 kJ per mole.) This value was subsequently confirmed by mass spectrometry. In the present work the enthalpies of combustion of the chloro, iso-butyl, and n-butyl derivatives of tetracyclopentadienyl uranium were determined and used to calculate their respective enthalpies of formation in both the crystalline and gaseous states. References 13: 8 Russian, 5 Western.

UDC 546.799.7

**Coordination Features of Tetravalent Berkelium and Its Isolation and Purification Based on This Method**

907M0118B Leningrad RADIOKHIMIYA in Russian  
Vol 31 No 6, Nov-Dec 89 (manuscript received  
27 Jun 89) pp 41-48

[Article by V. N. Kosyakov and N. G. Yakovlev]

[Abstract] The coordination behavior of the actinoid elements is closely related to their electron structure. The number of 5f-electrons determines the ionic radius which in turn determines the magnitude of the ionic potential, while the special features of the electronic configuration affect the participation of 5f-orbitals in the formation of chemical bonds. The tetravalent berkelium ion is one of the last available actinoid ions in the tetravalent state, i.e. an ion having the greatest number of 5f-electrons and the smallest ionic radius. In the present work an analysis of existing data on complex formation of actinoids and other tetravalent elements shows that as a result of its small ionic radius, tetravalent berkelium has coordination properties distinct from other tetravalent actinoids and Ce<sup>(IV)</sup>. This distinction is most clearly manifested during reaction with weak complex forming groups, such as NO<sub>3</sub><sup>-</sup>, Cl<sup>-</sup>, and CNS<sup>-</sup> which form chiefly extra-spherical complexes. This is supported by potentiometric data as well as data obtained while studying the mechanism of berkelium (IV) extraction from nitric acid solutions. The resulting information was used to develop new extraction-chromatographic methods for the isolation of Bk-249 from other actinoids and their fission products, including cerium, in a single cycle. Figures 6; references 31: 17 Russian, 14 Western.

UDC 546.799.4

**Reduction of Plutonium (IV) with Hydrazine in Presence of Solid Catalysts in Nitric Acid Solutions. Part I. Platinum on Silica Gel**

907M0118C Leningrad RADIOKHIMIYA in Russian  
Vol 31 No 6, Nov-Dec 89

[Article by I. G. Tananayev and V. P. Shilov]

[Abstract] The reduction of plutonium with hydrazine in acid solutions at temperatures below 40° C is a very slow reaction. It may be accelerated by using a heterogeneous catalyst, such as platinum on aluminum oxide or activated charcoal carrier. The study of other catalysts has both theoretical and practical merit. In the present work spectrophotometry was used to study the kinetics of plutonium (IV) reduction in nitric acid solutions in the presence of highly dispersed platinum on silica gel. It was demonstrated that this is an irreversible first order reaction in respect to the metal. The reaction rate constant is in direct proportion to the catalyst phase-to-solution ratio and the platinum-to-carrier ratio, and the concentration of the reducing agent is not a function of the H<sup>+</sup> ion concentration. The activation energy plutonium (IV) reduction was calculated to be 23 kJ per mole. Figures 3; references 3: 1 Russian, 2 Western.

UDC 542-61.62-278

**Study of Stability of Impregnated Liquid Extraction Membranes with Nuclear Filters**

907M0118D Leningrad RADIOKHIMIYA in Russian  
Vol 31 No 6, Nov-Dec 89 (manuscript received  
12 Dec 88) pp 100-104

[Article by V. P. Kapranchik, V. V. Proyayev, A. A. Kopyrin, and Yu. T. Slastennikov]

[Abstract] Selective transfer through an impregnated liquid extraction membrane is an effective method for separating mixtures of inorganic substances. This method has been used to great advantage in radioanalytical and radiotechnical work. Various porous materials such as polypropylene, fluorocarbon plastics, and nitrocellulose have been used as substrates, as well as nuclear filters. Advantages of the latter include precise determination of substrate characteristics, such as pore diameter, porosity, and thickness. The pore profile may be also be altered. Nuclear filters are mechanically, chemically, and radiationally stable and they are relatively cheap and available. However, their physical chemical stability is low. In the present work a study of various nuclear filter type liquid extraction membranes resulted in a new method for preparing these membranes from "double" filters. The procedure is simple and consists of applying the organic solution to the outside of the double filter. The solution then flows independently in the space between the filters. Figures 4; references 5: 2 Russian, 3 Western.

UDC 542.61:546.661:541.127

**Study of Kinetics of Di-2-Ethylhexylphosphoric Acid Induced Transfer of Europium Through Liquid Impregnated Membranes**

907M0118E Leningrad RADIOKHIMIYA in Russian  
Vol 31 No 6, Nov-Dec 89 (manuscript received  
2 Mar 89 pp 104-108)

[Article by M. N. Mikheyeva, A. P. Novikov, and B. F. Myasoyedov]

[Abstract] Membrane extraction is a promising method for separating substances from highly dilute solutions and precision refining and separation of elements, such as the radionuclides. However, industrial application of liquid membranes is encumbered with stability problems, such as gradual leaching of membrane liquids and the incumbent requirement for "supplemental feeding" of the matrix. This feeding does not always provide the desired results because transmembrane transfer is frequently associated with the formation of a stable microemulsion within the membrane. Leaching of an organic solution from the matrix increases with the pore diameter, polarity of the membrane phase, and the osmotic pressure. Since such matrix characteristics as thickness, pore diameter, and the matrix material itself, affect the stability of the membrane and its permeability, it becomes necessary to determine optimum properties. In the present work a study was made of the mass transfer kinetics of europium through di-2-ethylhexylphosphoric acid impregnated membranes of varying thickness and porosity. Mass transfer phase coefficients were calculated, and it was demonstrated that triple-layered matrices, where the thick porous inner film is surrounded on both sides with thin hydrophobic nuclear filters, have optimum properties. Figures 3; references 11: 5 Russian, 6 Western.

UDC 546.661

**Ion Exchange Separation of Macro Quantities of Europium (II) from Rare Earth and Transplutonium Elements**

907M0118F Leningrad RADIOKHIMIYA in Russian  
Vol 31 No 6, Nov-Dec 89 (manuscript received  
11 Apr 89) pp 109-113

[Article by G. S. Tikhomirova and L. I. Guseva]

[Abstract] The increasing demand for rare earth elements, particularly europium, in modern technology requires the development of simple and effective means for preparing europium compounds in high states of purity. While ion exchange appears to be one of the most effective methods, the separation of europium from other rare earth elements on cationites using oxycarboxylic or aminopolyacetic acids requires considerable expenditures in time and costly reagents. Preparation of highly pure europium compounds requires multi-fold separations from both rare earth and transplutonium

elements as well as organic complexes. Organic reagents are also low in resistance to radiation. In the present work a study was made of the possibility of separating europium in the di-valent state from tri-valent rare earth and transplutonium elements simultaneously by using cationites in moderately acidic solutions of hydrochloric acid in the presence of zinc amalgam as reducing agent. Satisfactory results were obtained with a single column containing Dowex-50X5, Zn(Hg), and 1 mole/liter HCl. Figures 2; references 14: 6 Russian, 8 Western.

UDC 546.110.23:547.15/17

**Tritium Labeling Natural and Modified Prostaglandins**

907M0118G Leningrad RADIOKHIMIYA in Russian  
Vol 31 No 6, Nov-Dec 89 (manuscript received  
30 Jan 89) pp 116-118

[Article by V. P. Shevchenko, V. V. Bezuglov, I. Yu. Nagayev, and N. F. Myasoyedov]

[Abstract] Direct introduction of tritium to natural and modified prostaglandins is an effective way to prepare radioactively labeled biologically active compounds. While natural prostaglandins with high molar radioactivity may be prepared by fermentation from polyene fatty acids labeled with tritium, modified prostaglandins can be prepared either by isotope exchange, selective hydrogenation, or selective dehalogenation. It is the specificity of biological and pharmacological action that makes preparation of both natural and modified tagged prostaglandins interesting. The most simple and widely used method for preparing multi-labeled prostaglandins is selective hydrogenation of the  $C_5=C_6$  bond by direct or selective reduction of various derivatives. In the present work a study was made of the conditions for introducing tritium using both methods. By using heterogenous catalysts, yields of up to 35 percent were attained. By using bulk substituents and  $(Ph_3P)_3RhCl$  as catalyst, up to 70 percent yield of labeled prostaglandin was achieved. References 13: 6 Russian, 7 Western.

UDC 546.110.23:547.15/17

**Synthesis of Tritium Labeled Arachidonic and Ekosapentaenic Acids Labeled at a Specified Double Bond**

907M0118H Leningrad RADIOKHIMIYA in Russian  
Vol 31 No 6, Nov-Dec 89 (manuscript received  
30 Jan 89) pp 119-120

[Article by V. P. Shevchenko, I. Yu. Nagayev, N. F. Myasoyedov, D. V. Kuklev, A. B. Imbs, N. A. Latyshev, and V. V. Bezuglov]

[Abstract] Success in contemporary research on eicosanoid metabolism is highly dependent on the preparation of labeled substrates having high molar radioactivity. One method for preparing such compounds

consists of selective reduction with tritium of monoacetylenic analogs, pre-synthesized from natural polyenic acids. Monoacetylenic analogs of arachidonic acid may be synthesized by two alternate methods. In the present work monoacetylenic analogs of arachidonic and eicosapentaenic acids were synthesized by selective and non-selective epoxidation of polyenic fatty acids. Oxidation of methyl arachidonate with *m*-chlorobenzoic acid resulted in a mixture of monoepoxy derivatives. Unless separated, the latter convert into the monoacetylenic analogs of arachidonic acid. After separation, the methyl esters were selectively reduced with gaseous tritium in the presence of Lindlar's catalyst. Corresponding polyenic acids were prepared in 50-60 percent yield from monoacetylenic analogs of arachidonic and eicosapentaenic acids. References 3: 1 Russian, 2 Western.

UDC 666.113.2:621.039.73

#### Glasses for Immobilization of Sulfate-Containing Radioactive Wastes

907M01181 Leningrad *RADIOKHIMIYA* in Russian  
Vol 31 No 6, Nov-Dec 89 (manuscript received  
4 Feb 89) pp 129-134

[Article by S. V. Stefanovskiy and F. A. Lifanov]

[Abstract] Glassification is one of the most effective means of treating radioactive waste for subsequent long-term storage or final disposal. The process is analogous to glass making, except that one of the components is radioactive waste. Large amounts of medium-level activity wastes are generated at nuclear power plants, radiochemical plants, and other such places. As a rule, these wastes consist of aqueous solutions or suspensions of sodium chloride and to a lesser extent chlorides of potassium, calcium, and iron. They are dried, mixed with boron silicate flux, and glassified at 1100-1200 °C. If the wastes contain heat resistant sodium compounds, such as sulfates or chlorides, the sulfate-silicate melts stratify, preventing formation of a homogeneous glass. Radionuclides of cesium and strontium, concentrated in the sulfate-chloride phase, are vaporized when this phase is broken at high temperatures. In the present work results are presented of research conducted at the Moscow NPO "Radon" on developing chemically stable glasses suitable for immobilization of sulfate-containing wastes of medium-level radioactivity. Alumino-, zinc phosphate, and borosilicovanadate glass systems, capable of dissolving large amounts of sulfate and nitrate radioactive wastes, were examined. Electron microscopy, EPR, and water resistance were used to study the structures of the glasses. The feasibility of making phosphate and borosilicate glasses containing 30-40 percent sulfate-containing wastes was demonstrated. Figures 6; references 7: 5 Russian, 2 Western.

UDC 546.799.3

#### Neptunium in Environment and Methods for Its Determination

907M0118J Leningrad *RADIOKHIMIYA* in Russian  
Vol 31 No 6, Nov-Dec 89 (manuscript received  
11 Jul 88) pp 134-142

[Article by Yu. Novikov, F. I. Pavlotskaya, and B. F. Myasoyedov]

[Abstract] In studying the behavior of heavy nucleides in the environment, most attention has been devoted to plutonium and to a lesser extent, americium and curium. The behavior of neptunium under natural conditions has been left almost not studied, owing to the low specific radioactivity of  $Np^{237}$  and the methodological difficulties in its determination. Work on the behavior of neptunium was confined chiefly to estimates of its radioactivity in natural sources (samples). However, study of the behavior of neptunium in the environment has great interest from the standpoints of the biogeochemistry and radioecology of this element, which has a greater mobility in eco systems than other transuranium elements. In the present work, known information on the content and behavior of neptunium in the environment, as well as present future methods for determining it, are discussed. Covered are sources from which neptunium enters the biosphere (underground nuclear testing, reactors, etc.); neptunium content in natural sources, chemical forms in which neptunium migrates into the biosphere; the behavior of neptunium in the environment; methods for detecting neptunium in natural sources. The amount of  $Np^{237}$  entering the biosphere, being commensurate in mass with that of plutonium, gives urgency to obtaining new data on the content neptunium in nature. Despite the lesser amount of radioactivity in the biosphere due to neptunium as compared to plutonium,  $Np^{237}$  may present greater potential danger owing to its greater migrating capability in ground water and soil. Figures 3; references 34: 6 Russian, 28 Western.

UDC 541.49:542.61

#### Coextraction of Nitrosoruthenium Nitrates with Uranium (IV) in System TBF-HNO<sub>3</sub>-UO<sub>2</sub>(NO<sub>3</sub>)<sub>2</sub>

907M0118K Leningrad *RADIOKHIMIYA* in Russian  
Vol 31 No 6, Nov-Dec 89 (manuscript received  
27 Jun 88) pp 143-147

[Article by A. V. Ochkin, A. V. Obruchnikov, V. S. Smelov, and V. V. Chubukov]

[Abstract] There are no references in the literature on the effects of uranyl-nitrate on the extraction of nitrosoruthenium nitrates with neutral organophosphorus compounds, particularly tri-*n*-butyl phosphate (TBF). However, it may be assumed that addition of uranyl-nitrate to nitric acid solutions of ruthenium complexes would have the same effect on the coefficient of distribution of ruthenium as with increasing the concentration of nitric



acid. Such an analogy is possible if the nitrosoruthenium nitrates are extracted with TBF not bound to solvates with nitric acid or uranyl-nitrate. In the present work a study was made of the effects of macroquantities of uranyl-nitrate on the extraction of nitrosoruthenium

nitrates from nitric acid media. Ruthenium compounds were detected in the nitric acid solutions that are capable of being extracted with TBF-HNO<sub>3</sub> solvate and co-extracted with uranyl-nitrate and TBF. Figures 2; references 7: 3 Russian, 4 Western.

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